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The Journal

of the

AMERICAN LEATHER CHEMISTS ASSOCIATION

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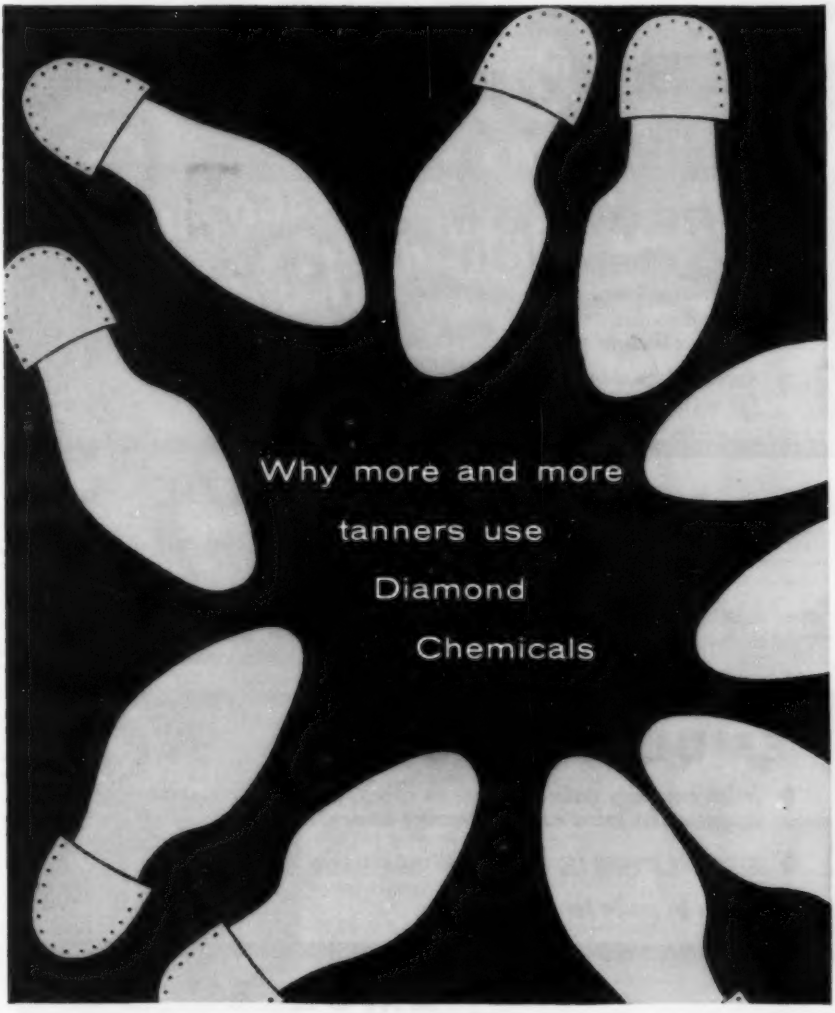

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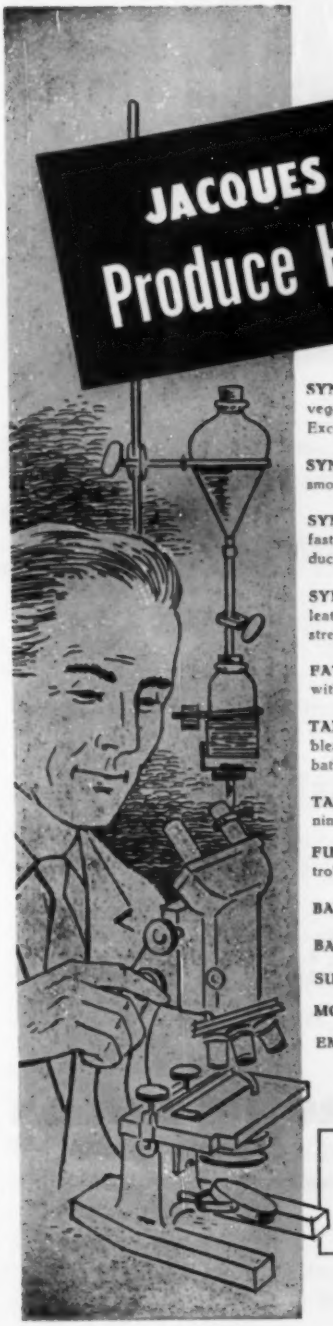

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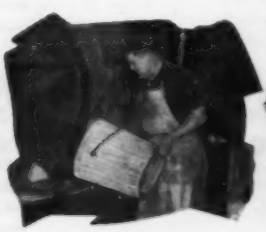

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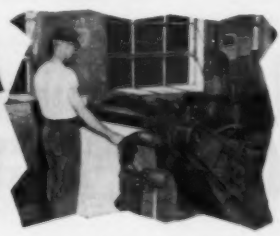

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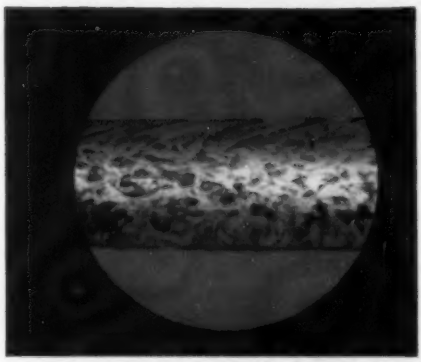

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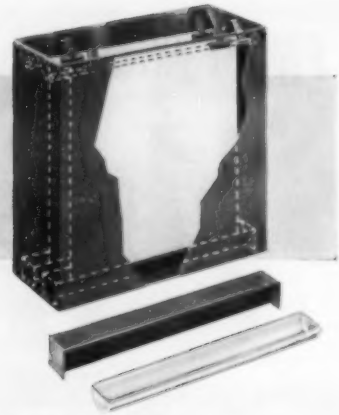

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AN IMPACT TEST FOR LEATHER BASED ON AN ALTERNATING PUNCHING FORCE

THOMAS J. CARTER

National Bureau of Standards
Washington, D. C.

ABSTRACT

An impact test based on the application of a punching force has been developed to evaluate shoe upper leather. A device delivers repeated thrusts to both sides of a specimen until failure occurs. The number of impacts required to produce cracking of the grain and puncture of the leather increases with increase in diameter of the plunger. The load per unit thickness of leather is inversely related to the number of impacts required to produce failure. Examination of the data obtained with the impact tester indicates that resistance to grain cracking, elongation, and puncture can be satisfactorily evaluated.

The usefulness of the machine has been investigated by determining the effects of lubricants, tannage, and accelerated aging. Straight chrome-tanned leather containing stuffing grease showed higher resistance to puncture on impacting than the similarly tanned leather containing fatliquor. When both leathers are degreased, their strengths are practically equal. This would suggest that the impact test can also be used to evaluate lubricants in shoe leather. Straight chrome-tanned leather showed much greater resistance to repeated impacting than either chrome syntan(Orotan)-retanned or chrome vegetable-retanned. The results obtained for the various tannages and for accelerated aging were found to be similar to those obtained in other fatigue tests.

INTRODUCTION

Leather is generally used in applications in which flexibility of the material is necessary. It is superior to most other materials in flexural endurance and for this reason is especially desirable for use in footwear. Investigations of the physical properties of leather have been intensified in recent years with special emphasis on those properties that pertain to the development of leathers for shoes.

The technique used to evaluate these properties has not been fully effective because of the lack of homogeneity in leathers. In testing leather to deter-

mine such properties as tearing strength, tensile strength, and elongation, tests are usually made in only one direction, either parallel or perpendicular to the backbone of the side. As most leather is used in the production of shoes, it is desirable that strength, elongation, and the tendency for leather to crack be evaluated by a procedure in which the results are independent of the direction of test. The impact tester provides such a method which is described fully in this report.

APPARATUS AND PROCEDURE

A general arrangement of the machine is shown schematically in Fig. 1, and a cross-sectional view with certain important dimensions is shown in Fig. 2. It consists of two hemispherically tipped plungers A_1 and A_2 which are attached to shafts mounted opposite to each other and on the same axis.

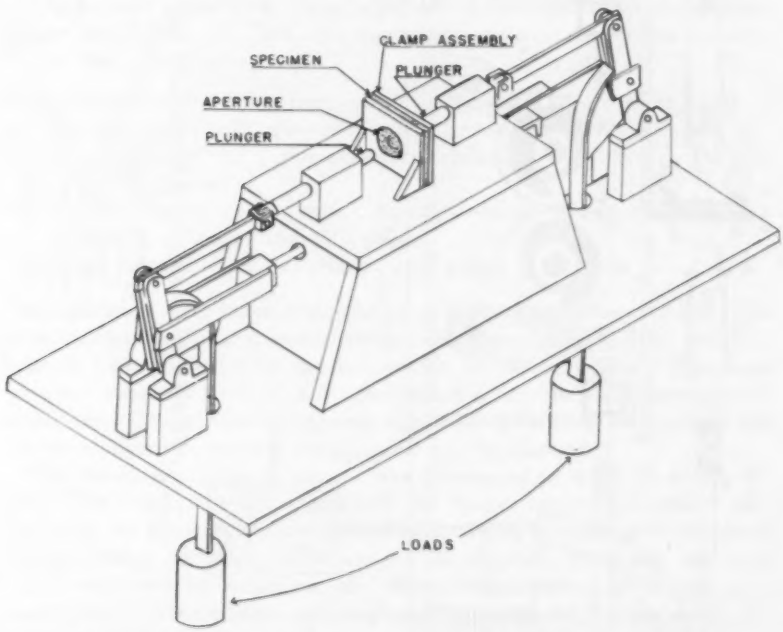

FIGURE 1.—Diagram of general arrangement of leather-testing machine.

The plungers are activated alternately by identical cams E_1 and E_2. The cam profile was designed so that the largest difference between the minimum and maximum radii is equal to 0.75″. These cams are driven in opposite directions of rotation through gears D_1 and D_2 by an electric motor. The

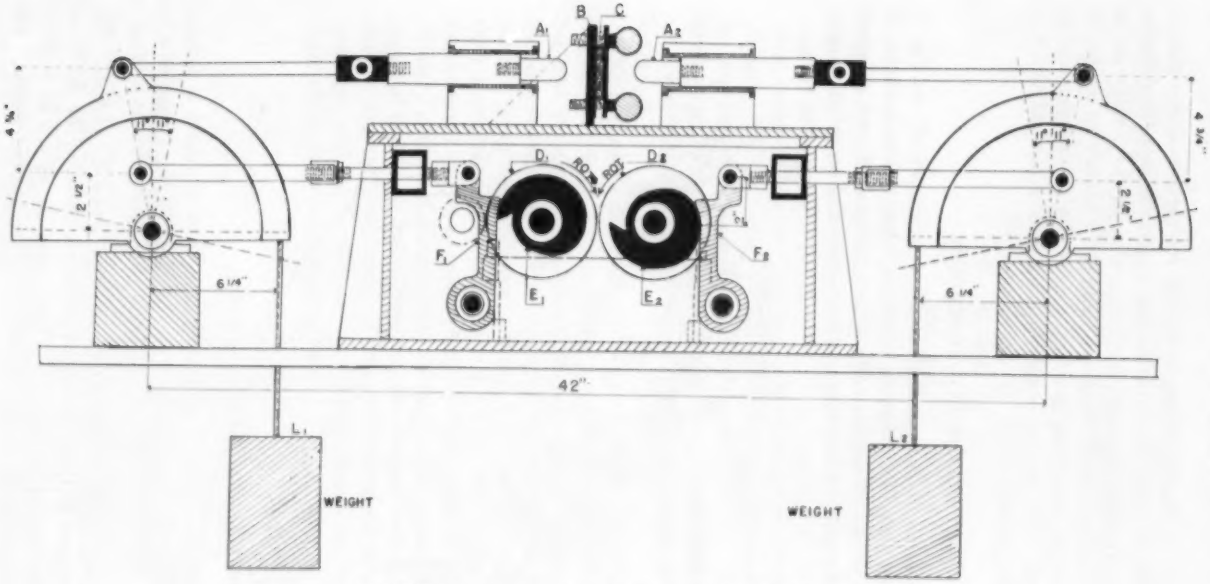

FIGURE 2.—Cross section of leather-testing machine.

A_1 and A_2—Plungers
B—Clamp assembly
C—Leather specimen
D_1 and D_2—Gears
E_1 and E_2—Cams
F_1 and F_2—Cam followers
L_1 and L_2—Loads

followers F_1 and F_2 control the reverse travel of the plungers. This is accomplished by contact with the follower and the cam, and by the increase in radii as the cam rotates. Upon reaching the indented part of the cam the follower releases the plunger which is then activated by the load L_1 or the corresponding load L_2. The force applied to the specimen is supplied by the load acting through linkages as shown in Fig. 2. The specimen clamp assembly consists of two serrated steel templates, and its over-all dimensions are approximately 6″ x 4.5″ x 0.75″. The aperture through which the plungers pass is 1.5″ in diameter. The specimen C is held in an upright position, and impacts are delivered alternately to both sides.

Unless otherwise stated, a 27-lb. load, plungers of 0.50″ diameter, and 22 cycles (44 impacts) per minute were employed in the test. The diameter of the aperture and the rate at which the load is applied to the specimen were arbitrarily chosen and were not varied.

The leathers tested were of the USXL series prepared under the sponsorship of the OQMG (1). The following treatments were used in the preparation of the leathers tested:

(*a*) Chrome only—4.5% glucose-reduced chrome and 25% stuffing grease.
(*b*) Chrome only—4.5% glucose-reduced chrome and 9–14% fatliquor.
(*c*) Chrome retan—4.5% glucose-reduced chrome and 40% syntan (Orotan) plus 25% grease.
(*d*) Chrome retan—6.5% sulfur dioxide–reduced and formate chrome plus 20% retannage and 25% grease.
(*e*) Vegetable only—regular process, 25% grease.

The specimens were taken from the same location of different sides. The samples were conditioned several weeks in an atmosphere of 70°F. and 50% relative humidity prior to the preparation of the specimens. Specimens were cut approximately 4″ x 4″ and tested under the above atmospheric conditions. The number of impacts required to produce grain cracking and for the plunger to penetrate the specimen was determined.

The elongation of the specimens was determined at intervals during the test. The machine was stopped with the leather specimen extended fully by one of the plungers and the amount of extension determined at the center of the leather specimen. The amount of specimen extension was most easily evaluated by measuring the plunger displacement, using two arbitrarily fixed reference points on the plunger assembly for this purpose.

RESULTS AND DISCUSSION

The effects of thickness of leather and diameter of plunger on the performance of the machine were studied. The usefulness of the machine in evaluating leathers was investigated by studying the effects of different tan-

nages and accelerated aging on the resistance of leathers to the punching force. The influence of lubricants is also shown.

Effect of thickness of leather.—The effect of thickness of leather on resistance to impact was studied with specimens of straight chrome-tanned leather containing grease and of thicknesses ranging from 94 to 30 mils. The following formula was used to calculate an arbitrary loading factor per unit thickness:

$$\text{lb/100 mils thickness} = \frac{27}{\text{thickness of specimen in mils}} \times 100$$

The calculated loads ranged from 29 to 90 pounds. A plot of the logarithm of the number of impacts to failure versus the logarithm of the number of pounds per unit thickness is shown in Fig. 3. The straight line shown was fitted to the data by the method of least squares. It indicates that the load per unit thickness is inversely related to the number of impacts required to produce failure.

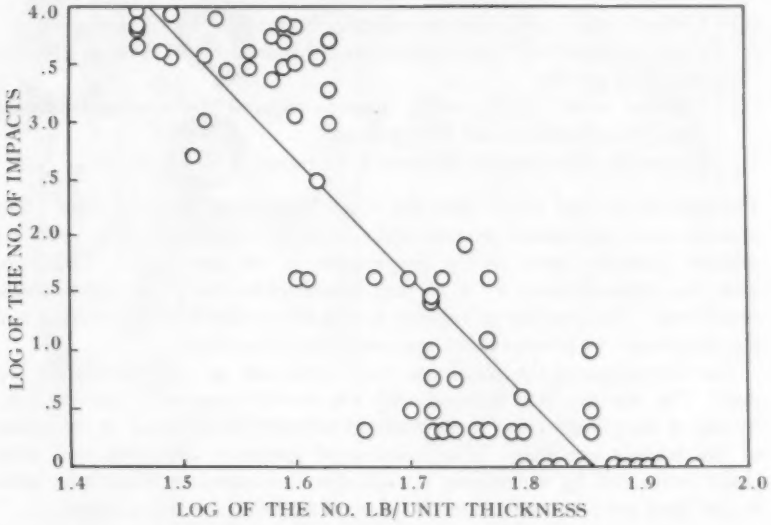

FIGURE 3.—Relation of load per unit thickness of leather and resistance to impact. Plunger diameter ½".

Effect of diameter of plunger.—The effect of diameter of plunger on the resistance of leather to grain cracking and to puncture was studied with plungers ranging from 0.25" to 1.0" in diameter. The largest diameter

used for puncturing the leather was 0.850″. The leathers tested included chrome-tanned, chrome syntan (Orotan)-retanned, chrome vegetable-retanned, and straight vegetable-tanned. Crackiness of the grain was observed at intervals with a magnifying glass while the machine was in operation and also while stopped with the plunger exerting full load on the flesh side of the specimen.

TABLE I

EFFECT OF PLUNGER DIAMETER ON GRAIN CRACKING AND ELONGATION OF CHROME-TANNED LEATHER*

	Chrome-tanned Leather			Chrome Vegetable-tanned Leather					
	Containing Grease			Containing Grease			Degreased		
Diameter, inch	Thickness, mils	No. of impacts to cause grain cracking	Elongation, %†	Thickness, mils	No. of impacts to cause grain cracking	Elongation, %†	Thickness, mils	No. of impacts to cause grain cracking	Elongation, %†
1.000	95	13443	13.3	86	3900	13.3	80	2766	15.0
0.875	89	5647	15.7	84	1862	13.3	82	957	18.0
0.750	94	3800	15.0	84	1527	15.0	86	385	15.8
0.625	97	108	15.3	85	1210	16.0	85	153	18.0
0.500	96	74	16.7	87	1	17.5	88	16	19.0
0.375	98	8	16.7	84	1	17.0	85	1	21.5
0.250	93	1	16.0	82	1	18.0	86	1	22.3

*The values represent averages of 3 to 4 measurements.
†The test was continued 1 minute after the appearance of the first visible grain crack.

Table I shows that in general the differences in resistance to grain cracking obtained with diameters ranging from 0.25″ to 0.50″ are not significant. However, with diameters 0.50″ to 1.0″ the number of impacts necessary to produce initial cracking increased with increase in diameter. The results also show that the elongation of the leather increased with decrease in diameter of plunger.

In this respect these findings are in good agreement with those obtained in a study of a burst test applied to shoe upper leather (2). However, the impact test, when used to evaluate grain cracking, is much more sensitive to size of diameter of plunger.

Figure 4 shows the effect of plunger diameter on the resistance of leather to puncture. The logarithm of the number of impacts versus the reciprocal of the diameter of plunger can be reasonably represented by a straight line relation.

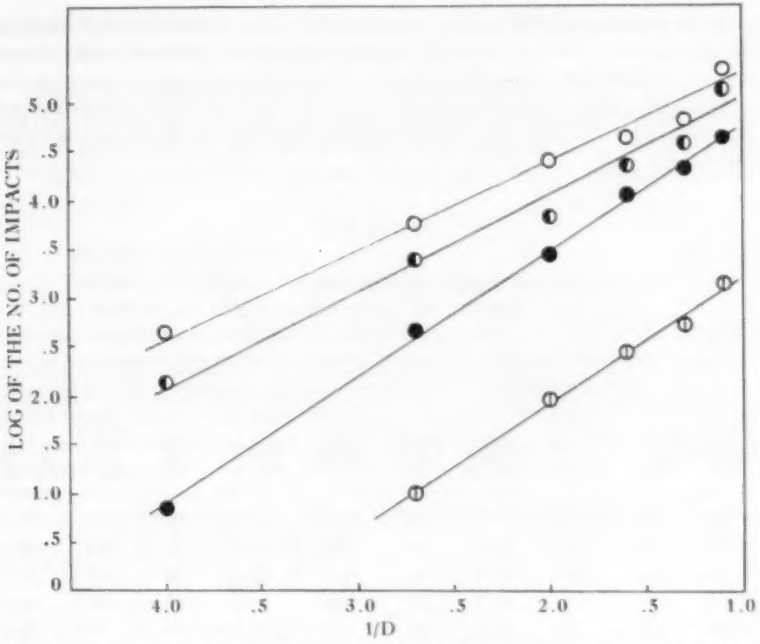

FIGURE 4.—Relation of diameter of plunger and endurance; 1/D = reciprocal of diameter of plunger; ○ straight chrome-tanned; ◐ chrome Orotan-retanned; ● chrome vegetable-retanned; ⓘ straight vegetable-tanned.

Effect of tannage and accelerated aging.—Leathers of different tannages, with and without lubricants, were aged at temperatures of 80°, 100°, and 120° C. in air for 96 hours and then conditioned in an atmosphere of 50% relative humidity and a temperature of 72° F. for one week before testing. The results, summarized in Table II, show that among the unaged lubricated leathers the resistance to impact is greatest for the chrome-tanned leather containing stuffing grease and lowest for the chrome vegetable-retanned leather. The fatliquored chrome and the stuffed chrome Orotan-retanned leathers are very nearly equal in strength and are intermediate.

The importance of lubrication is shown by the comparatively low resistance of the degreased leathers. Among this group of unaged degreased leathers the two chrome-tanned leathers are practically equal in strength and are superior to both chrome Orotan-retanned and chrome vegetable-retanned leathers. The values obtained for each of the leathers described rank in the same order as those for another fatigue test which is based on flex tension (3). Comparison of the endurance of leathers as determined with the impact machine and a flex tension machine is shown in Fig. 5.

TABLE II

EFFECT OF TANNAGE AND ACCELERATED AGING ON RESISTANCE OF LEATHER TO IMPACT

Tannage	Number of Impacts Required to Puncture the Leather				Ratio A/U†		
	Unaged	Aged					
		80°C.	100°C.	120°C.	80°C.	100°C.	120°C.
Chrome, containing stuffing mixture*	29040	12144	6952	2	0.42	0.24	0.00
Degreased	1694	1848	1650	946	1.09	0.97	0.56
Chrome, containing fatliquor*	16412	16148	7876	344	0.98	0.49	0.02
Degreased	1749	1870	1408	14	1.07	0.81	0.01
Chrome syntan (Orotan)-retanned, containing stuffing grease	16324	13904	11110	1342	0.85	0.68	0.08
Degreased	594	550	682	550	0.93	1.15	0.93
Chrome vegetable-retanned, containing stuffing grease*	9812	1936	1234	374	0.20	0.13	0.04
Degreased	440	638	512	35	1.45	1.30	0.08

*Original leather containing grease.
†Expressed as the ratio of the average value for aged leather to the average value for the unaged leather.

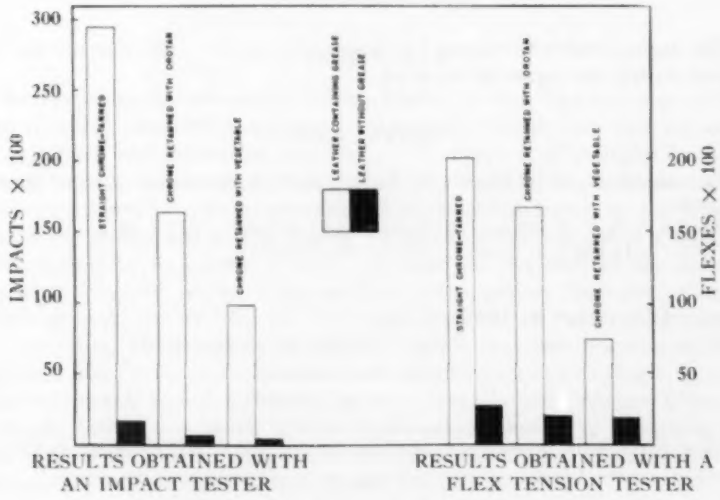

FIGURE 5.—Endurance of typical shoe upper leather.

The results for the aged leathers given in Table II indicate that the leathers containing grease were more susceptible to deterioration on aging than the degreased leathers. Judging from the loss in strength due to aging of the stuffed chrome-tanned and chrome vegetable-retanned leathers, it appears that their critical temperature is appreciably below 100° C. In contrast to these leathers the fatliquored chrome-tanned and the stuffed chrome Orotan-retanned leathers show considerably less deterioration at 80° C. However, between 100° and 120° C. all the leathers, except the degreased chrome Orotan-retanned leather, showed marked deterioration regardless of the type of lubricant or tannage.

SUMMARY

An impact test based on an alternating punching force is described. It has the advantage of eliminating directional effects that are commonly encountered when strength measurements are made in one or two directions only, either parallel or perpendicular with respect to the backbone line. The results obtained in the impact test show that differences in the rate of grain cracking, elongation, and the complete breakdown of the leather were caused by varying the diameters of the plungers. Differences in endurance caused by thickness of leather, lubricants, tannage, and accelerated aging were found to be similar to those found in other types of fatigue tests.

ACKNOWLEDGMENT

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United States cattle imports rose sharply in the first half of 1957 compared with 1956. Increased imports of feeder cattle from Mexico accounted for most of the increase.

THE QUESTION OF THE EXISTENCE OF RETICULIN IN ANIMAL SKIN*

A Critical Survey of Recent Literature

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ABSTRACT

In the corium region of the skin there are two types of connective tissue—collagen and elastin—the difference being mainly histochemical. A third type of connective tissue, "reticulin", does not exist. Those parts of the skin which have been defined as "reticular tissues" are in fact collagen of a variant morphology. The introduction of the words "reticulin" or "reticular tissue" into the description of the connective tissue of the skin has recently given rise to considerable confusion—reticulin being mistaken for elastin or for soluble collagen (procollagen). The object of this paper is to clear up and correct these mistakes.

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INTRODUCTION

Anyone who reads attentively the more recent publications on the structure and chemical constitution of skin will be struck by the fact that reticulin is mentioned with increasing frequency. The expression "reticulin" can be traced back to the American anatomist F. E. Mall (1), who gave the name "reticular tissue" to the coarse-meshed, networklike tissue in the lymph nodes, the spleen, and similar organs. This tissue reminds one of a sponge, with respect to its coarse porosity. A cross section through the spleen, lymph glands, etc., shows a picture that differs greatly from the collagen weave of skin. Apart from the open-mesh structure of the reticular tissue, its individual fibers cannot be resolved under the light microscope into subfibers and fibrils, as in the case of the relatively compact collagen weave. Another morphological difference between reticulin and collagen fibers is that the former apparently exhibit anastomosis, that is, a branching or reuniting of the fibers, while the latter never exhibit branching except to the extent that fiber bundles may divide into part bundles.

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In 1929 the British histologist M. Kaye (2) propounded the thesis that reticulin exists also in animal skin and suggested that the grain membrane, as well as the loose sheath network that encloses the collagen fiber bundles with a network of very fine fibers, is made up of this type of tissue. Later, O'Flaherty and Roddy (3) designated as reticulin the connective tissue framework of the subcutaneous fatty tissue.

However, the application of the expressions "reticulin" or "reticular tissue" to certain structures of animal skin—expressions used originally only in the literature of leather science—has always been restricted to that compartment of scientific literature. Medical, veterinary medical, and zoological textbooks of histology and anatomy have taken no notice of this innovation. If one consults the most authoritative technical literature in the field of histology, one learns that human skin contains only two connective tissues, namely, collagen and elastin. Even within the technical literature of leather the reticulin hypothesis has been challenged insofar as it designates connective tissue structures of skin, since good arguments have been put forward showing that both the grain membrane and the loose sheath tissue of the hide fiber systems are merely special morphological forms of collagenous fiber structures (4). These arguments have not, thus far, been refuted. Nevertheless, the expression "reticulin" bobs up continually in publications in the field of leather science, often used in connections that would lead one to believe that a clear understanding has been lost as to what the term really means. The resulting confusion of ideas makes this explanatory statement necessary.

As will be shown, three types of uncertainties or changed meanings have arisen from the use of the words "reticulin" or "reticular tissue" in the leather literature:

(*a*) No sharp distinction has been drawn between the morphological and the chemical meaning of these terms.

(*b*) Reticulin frequently is confused with elastin, and vice versa.

(*c*) Reticulin has also been regarded as synonomous with procollagen or precollagen, whereby confusion arises because procollagen is used also as a term for soluble collagen.

These sources of confusion and uncertainty will be discussed and corrected in the following paper. In conclusion new investigations in the field of reticulin will be reported.

VAGUE DISTINCTION BETWEEN THE MORPHOLOGICAL AND THE CHEMICAL MEANING OF THE CONCEPT "RETICULIN"

From the outset, the concept "reticulin" was plagued with a double meaning inasmuch as this word sometimes implied a *morphological* distinction

between collagen and reticulin, while at other times a *chemical* difference between these two types of connective tissue was implied. Because the morphological and chemical aspects were not sharply differentiated, there arose the iridescent vagueness of the word "reticulin", which Wassermann (5), one of the best authorities on connective tissue structures, has characterized briefly as follows: "The name 'reticulin', since it was introduced, has acquired a certain double significance."

The morphological differences between collagen and the reticular tissues in the spleen, lymph glands, kidney wall, etc., have already been mentioned (different tissue density, and differences in respect to branching). It should be added that collagenous fibers, in contrast to those of reticular tissue, show double refraction. In this connection it must be noted that the connective tissue structures of the skin, designated by Kaye as reticulin, are anisotropic, i.e., show double refraction. This by itself should have been enough to classify these tissues as collagen and not as reticulin.

As concerns the histochemical characteristics of reticulin and collagen, the following are the chief differences that are emphasized in the literature covering the subject:

Nature of Test	Collagen	Reticulin (from spleen, etc.)
Staining with acid fuchsin and picric acid according to van Gison	Yellow-red	Weak bluish red
Staining by silver impregnation	Weak yellow-brown	Dark black

These chemical differences have led to the assumption that the reticular tissue is composed of a special protein, namely "reticulin".

The staining of skin sections with acid fuchsin–picric acid, which Mall, the discoverer of reticulin, gave as a criterion for reticular tissue, has been used by Kaye (6) as evidence for the presence of reticular tissue in skin. In skin sections thus stained the large collagen fibers appear bright yellow-red, while the finer fibrils, including those of the loose fiber sheath tissue, are stained bluish red. This color difference is not, however, caused by chemical difference, but rather by a difference in density between the two kinds of fibers, as was shown by Küntzel and Seitz (4). If a skin fiber is frayed out with a needle into fibrils, the isolated fibrils assume the same bluish-red color as reticulin does on treatment with acid fuchsin–picric acid. One can readily obtain specimens in which one side of a fiber bundle has the typical yellow-red color of collagen, while the other side, after separation into fibrils, possesses a bluish-red color. Furthermore it is possible to bring about a color change from bright yellow to bluish red simply by opening up into fibrils a previously stained skin section. From this it follows that the difference in

color does not originate from different rates of diffusion of the dye through a compact tissue as contrasted with a frayed-out, loosely woven connective tissue; rather, the color difference when the individual fiber elements are stained uniformly depends solely on the density, i.e., the compactness, of the fibers when they are observed under the microscope.

Regarding the silver impregnation*, the reticulin fibers are known also as "argyrophilic fibers" because of the intense blackening by deposition of silver granules. As will be discussed later in more detail, collagen fibers pass through an embryonic stage in which they too can be more strongly impregnated with silver and manifest an enhanced argyrophilia. This has led to the assertion that reticulin fibers are precursors of collagen.

In spite of the morphological and histochemical differences, both sorts of tissue behave the same toward enzymes, as shown by Kramer and Little (9); neither is attached by trypsin or by hyaluronidase, and both are dissolved by pepsin or by the collagenase of *Clostritium welchii*. In contradiction to this statement Kaye claimed that the alleged reticulin of the skin, particularly the fiber sheath, possessed a certain inertness toward enzymes that could be used to differentiate reticulin from collagen, but Küntzel and Seitz (4) showed that the fiber sheath is neither more nor less resistant to enzymes than the fiber itself, so that susceptibility to enzymic action affords no ground for assuming that the two tissues are different chemically.

This dual significance of the word "reticulin", morphological and chemical, doubtless is responsible for the fact that the term has been applied to skin structures to which the original definition of reticulin cannot possible apply. There do exist, indeed, morphological but not characteristic chemical differences between typical skin collagen on the one hand and the (supposed) skin reticulin on the other. The word "reticulin" has been used to give a specific designation to these differences, without paying enough attention to the secondary meaning of the word.

EXAMPLES OF CONFUSION OF RETICULIN WITH ELASTIN

A remark of Stubbings, made in the course of a recent discussion of "The Network Structure of Elastin in the Grain Layer of Cattle Hide" (10) is characteristic of the prevailing uncertainty regarding the relation between elastin and reticulin. He said: "It has always been very confusing to me whether or not this so-called reticulin is the same as elastin, or whether they are two separate and distinct things".

The older literature also contains cases of confusion between loose sheath tissue, designated as reticulin by Kaye, and the elastin fiber system. This

*The technique of silver impregnation consists of first tanning the connective tissue structure with formaldehyde, then treating it with an oxidizing agent (permanganate or hydrogen peroxide), next soaking it in ammoniacal silver hydroxide, and finally reducing the silver salt with formaldehyde. The general procedure is susceptible to countless variations. According to Dempsey, Haines, and Hitchborn (7) the modified method of N. C. Foot (8) is best suited for differentiating collagen and reticulin.

confusion evidently came about because collagen fibers swell strongly in acid or alkali, while the fiber sheath material apparently does not, since it creates small, ringlike constrictions in the swollen fiber. Schaefer, in Quain's "Elements of Anatomy" (cited by A. Seymour-Jones [11]) gives a description of the loose connective tissue of the corium (called "areolar tissue" by the British anatomists) and states that this tissue consists of elastin fibers which, being themselves incapable of swelling, form rings about the swollen collagen fibers. This assumption cannot, however, be correct, since the constricting fibers do not stain like elastin. A. Seymour-Jones proposed a modification of Schaefer's view, whereby the "areolar fibers" were referred to as "white elastic fibers", that is something intermediate between collagen and elastin. Later, Kaye tried to explain the facts of the constriction of swollen collagen by (apparently) nonswollen sheath fibers by means of the reticulin hypothesis; the constricting sheath fibers were supposed to be composed of reticulin, which was known not to swell like collagen in acids or alkalies. It is superfluous, however, to assign a different chemical constitution to this sheath tissue, because it really swells exactly like the main part of the collagen, as one can demonstrate especially well in the case of the subcutaneous tissue that corresponds to the sheath tissue. A short treatment with alkali or acid transforms this subcutaneous tissue to a strongly swollen, slimy, gelatinous mass. The fact that the sheath fibers that surround the fiber bundles in the form of rings apparently do not swell is due to the peculiarity that collagen swells in acid or alkali only when it can contract in the direction of the fiber axis (12). Because the volume of the fiber bundle that is surrounded by the sheath increases markedly on swelling, the enveloping sheath system is stretched strongly, and consequently cannot swell, since the tension prevents it from contracting in the direction of the fiber axis. If the collagen fiber is cut at a constriction, and the constricting sheath system is then isolated, it will be found that the latter swells to an amorphous gelatinous state.

The grain membrane, described by Kaye as reticulin, likewise has not escaped the fate of being referred to as elastin. Not long ago, four American authors (13) hydrolyzed a piece of steerhide in water alone at elevated pressure (23 lb. per sq. in.), whereby they obtained a fine-fibered membrane whose fibers showed the characteristics of elastin. This membrane was erroneously described by these authors as the "grain membrane", that is, the uppermost zone of the papillary layer. Thus we find in the literature three different assertions about the composition of the grain membrane. The common assumption is that the grain membrane is composed of collagen, because collagen fibers of the lower skin layers continue uninterruptedly into the grain membrane. This is the author's conception of the case. Kaye designated the material of the grain membrane as reticulin. And now, finally, we have the grain membrane equated with elastin! Happily these

American authors soon set matters right by announcing that not merely the grain membrane is set free by hydrolyzing collagen in the autoclave but rather the whole elastin fiber system, which permeates the whole papillary layer and attains its greatest density in the middle of this layer, at about the level of the fat glands (14).

This mixing up of elastin with reticulin has received another push forward in that Bose and Das have recently used the method given by Kaye for isolating reticulin (boiling the skin material for 4 hr. in 0.05*N* NaOH) to isolate the alleged reticulin from skin (15). As long ago as 1936 Küntzel and Seitz showed that the residue obtained by cooking animal hide by Kaye's method is really elastin because it reacts with elastin stains in the characteristic manner. It is perhaps understandable that these Indian authors might not know of this German language publication of 1936, although the paper in question was indexed in the English language technical literature under the heading of "reticulin" (16). It is, however, surprising that Bose and Das accepted uncritically the alkaline hydrolysis method for isolating alleged reticulin and were not aware that other authors (17) had, shortly before, published work in the same journal wherein the residue obtained by alkaline hydrolysis of the same kind of material at elevated temperature was considered to be elastin.

Bose and Das set themselves the task of separating the proteins of Indian hides into fractions, so far as possible, and then ascertaining the amino acid makeup of the individual fractions. In fractionating the proteins these Indian authors went much farther than workers with similar objectives, such as Jacobs (18), Stubbings (17), and DeBeukelaer (19), in that they wanted to separate the skin proteins into eight fractions: the fibrous proteins collagen, reticulin, elastin, and keratin, and the nonfibrous proteins albumin, globulin, mucin, and myosin. It is to be noted that none of the other authors just named tried to isolate reticulin.

Since the "reticulin" isolated by the method of Kaye, which Bose and Das adopted without checking, actually is elastin, one asks next where the elastin fraction obtained by these authors could have come from. The answer is simple: Bose and Das obtained elastin from another organ, namely the neck ligament of cattle! One might then expect that somewhat the same amino acid constitution would be found for the alleged reticulin of the skin (really elastin) and for the neck ligament elastin. This is however by no means the case (see Table II of the Bose-Das paper, *JALCA*, **51,** 652 [1956]). The discrepancy between the two series of figures is explainable by the fact that neck ligament elastin is contaminated by considerable quantities of collagen (20). On the other hand, the hide elastin was doubtless strongly attacked during the drastic preparation method of boiling with alkali and very likely underwent a change in amino acid composition. At any rate one could not expect, knowing the procedure that was used, that materials so different in

origin and treated so differently would show the same amino acid composition.

Bowes and Kenten (20) in 1949 determined the amino acid constituents of elastin of the neck ligament and of skin elastin by qualitative paper chromatography. The skin elastin was liberated by autoclaving with water and by boiling for 15 minutes in 0.01*N* NaOH. The elastin prepared from skin showed important differences in amino acid makeup from neck ligament elastin, so that these authors discussed the possibility that these two forms of elastin are, in nature, constituted differently. However, the question has not been studied as to whether neck ligament elastin would be altered in the same way with respect to amino acid makeup, if it were treated by the procedure used to obtain skin elastin.

CONFUSION OF RETICULIN WITH SOLUBLE COLLAGEN ("PROCOLLAGEN")

Because reticulin, that is, the reticular tissue of the spleen, etc., manifests the histological characteristic of argyrophilia (capability of being intensely impregnated with silver granules), and since embryonic collagen likewise is more argyrophilic than the mature collagen fibers with which the tanner usually has to deal, the idea has arisen that reticulin is the same thing as embryonic collagen (21). In that connection reticulin has been described in the literature, with the special meaning of an embryonic precursor of collagen, as "procollagen" or "precollagen". Thus Gustavson writes in his recent book *The Chemistry and Reactivity of Collagen*: "Reticulin represents an early stage in the development of collagen; it is procollagen".* O'Flaherty stated, in the discussion already referred to (10), concerning the network structure of elastin in the grain layer of steer hide: "As an older histologist I would talk about reticular tissue; today I would talk about precollagen as a material rather than as reticular tissue. It is merely a precursor of collagen rather than as distinct entity."

Contrariwise, in the field of collagen investigation procollagen or precollagen means something altogether different, namely, a *soluble form of mature collagen* which can be extracted from hide or tendon collagen with very dilute organic acids (23). It is obvious that in this case procollagen means something altogether different from reticular tissue. Reticular tissue is recognizable to the eye, by histological examination, under low magnification, as a definite structure of morphological identity, though only in certain specified organs, of which the skin is not one. The soluble fraction of collagen, known as procollagen, cannot be recognized by microhistological techniques amid the insoluble portion of mature collagen. Native collagen looks no different under the microscope than collagen from which the soluble fraction has been removed by treatment with citrate buffer and the like. Procollagen,

*Actually Gustavson merely mentioned this as being one of at least four different opinions. He went on to say that "the concept of reticulin as a collagen type of protein modified by association with polysaccharide seems to be the most acceptable." See *Das Leder*, **8**, 144–45 (1957).

in the sense of soluble collagen, can be made visible only by extracting it from the mature tissue and then regenerating it in fibrillar form by dialyzing the extract.

The "procollagen" which is soluble in dilute organic acids received its name from the Russian collagen researcher Orekhovich (24) because originally it was looked upon as the building material from which the collagen fibers (which undergo a continuous interchange of materials like any other living tissues) are regenerated. This assumption has not been confirmed. Harkness and his coworkers (25) have shown that it is not the acid-soluble form of collagen but rather that which dissolves in a neutral or weakly alkaline environment that plays a role in fibrogenesis and thus perhaps ought to be designated as procollagen.

On these and other grounds, Highberger, in his great review chapter on the chemical structure and macromolecular organization of skin proteins (26), proposed that the term "procollagen", as applied to soluble collagen, should be abandoned altogether. The term "tropocollagen" was suggested as more appropriate. Tropocollagen as the designation of soluble collagen emphasizes the fact that the latter can be recovered by recrystallization as fibrillar structures showing various periodicities in electron microscope pictures, or as fragments of fibrils (so-called segments). It is to be hoped that Highberger's proposal will receive general consideration, to put an end to the confusion regarding the meaning of "procollagen".* At all events, the equating of pro- or precollagen with reticulin is to be avoided, not merely on semantic grounds but also for more objective reasons, as will be shown in the next section.

THE PLACE OF RETICULIN TISSUE WITHIN THE CONNECTIVE TISSUE TYPE GROUP

Kramer and Little (9) have recently succeeded in separating the reticular tissue from the kidney wall by maceration with 0.1*N* NaOH or by intensive tryptic digestion, in a condition such that electron microscope pictures of the residual tissue can be obtained. Metal-shadowed pictures reveal this reticulin as a mat of fine, interwoven collagen fibrils imbedded in an obviously amorphous ground substance. The fibrils vary in diameter from 400–600 A. down to less than 100 A. The most important fact revealed by these electron microscope pictures is the cross-banding of these fibrils, with a periodicity of

* *Note added by Dr. Küntzel.*—At least three kinds of soluble collagen have been described in the literature —the alkali-soluble, the acid-soluble, and the neutral salt-soluble. Moreover the Russian school persists in retaining the name procollagen for the acid (i.e., citrate buffer)–soluble fraction although this fraction is less well qualified for this appellation than are the other types of extractable collagen. For these reasons it seems advisable to avoid, and if possible do away with, the use of the terms "procollagen" and "precollagen". Instead, these fractions ought to be designated according to their methods of preparation. Even the expression "tropocollagen", suggested by Highberger as a collective name for certain types of soluble collagen, seems to me to be premature at present. At the International Symposium on Connective Tissues held in London in June 1956 under the auspices of the International Organizations of Medical Sciences a committee was formed to take up the question of the nomenclature of soluble collagen. —A.K.

the order of magnitude of 640 A., *which shows that these fibrils are identical with collagen.*

Two conclusions follow from this research:

(*a*) Reticulin fibers are not composed of one homogenous substance but consist of submicroscopic fibrils and a larger proportion of an interfibrillar amorphous cementing substance. This cementing substance is notable for its high content of glycoproteins (mucoids), which explains the intensive staining of reticular tissue by silver impregnation.

(*b*) The fibrillar portion of the reticular fibers consists of collagen, confirming the close relationship between reticulin and collagen.

These findings take on special significance in the light of a comparative study of different connective tissue structures that Schwarz (27) has recently carried out. According to this study all kinds of connective tissue can be assigned a composition consisting of a mixture of fibrillar material and amorphous cementing substance, only the proportions of the two components being variable. Furthermore, the ratio of fibrillar to amorphous substance varies during the course of development from the embryonic precursor form to the "terminal" structure.

For example, the embryonic precursor of collagen is rich in amorphous material. With increasing age of the collagen fibers, the amorphous material decreases in amount, this being associated with a multiplication and increase in density of the fibrils. In fully developed tendons, or the fiber bundles of the corium, it is generally not possible to demonstrate the presence of mucoids (characteristic of cementing substances) by histological techniques; this means that in collagen the fibrillar fraction predominates, this fraction being characterized by showing double refraction in polarized light and an X-ray "fiber diagram."

A change in the manner of silver uptake goes hand in hand with the growth of the fibrils. In young, immature collagen silver is deposited only on the outer surface of the fibrils, and the individual silver grains are irregularly distributed. This silver deposition becomes increasingly regular with increasing age. First the silver granules are deposited in a periodic manner on the outer surface, and then, as development proceeds they are deposited inside the fibrils, in the so-called "D-regions" of the cross bands. This brings out more strongly the cross-banding that is detectable under the electron microscope. The terminal stage of fibril differentiation is reached only when, on staining with silver, the silver granules are deposited periodically within the fibril in accordance with cross-banding. The completely differentiated fibrils, stained with silver under specified conditions, present a histological picture of being stained brown, while the preceding stages, conditioned by the abundance of mucoid in the more abundantly present cementing substance, are stained more intensively, becoming violet or even black.

Schwarz, from whose researches on the "Interstitial Substances of Connective Tissue" the preceding line of thought has been taken, does not believe that collagen is sufficiently distinguished from reticulin and other incompletely developed connective tissue structures by the cross-banding that is visible under the electron microscope, since this cross-banding is characteristic also of the fibrils of other connective tissues such as those of reticulin and elastin. As a differentiating mark of recognition, the interior silver staining of the fibrils is much more applicable to the characterization of mature collagen.

As Link (28) has established, the different connective tissue structures of the animal or human body reach the final stages of their developments with differing speeds. For example, the collagen fibrils of the skins are already completely differentiated during intrauterine life, while tendon collagen reaches this stage only during the first years after birth. The cornea of the eye remains permanently in a still earlier stage of development. Schwarz writes concerning this: "In the cornea there are to be found fibrils that can only be described as embryonic, with respect to their state of development. They show an irregular surface staining with silver, which collagenous tissue shows only in the embryonic stage. If one attempts to follow the evolution of the cornea from the earlier embryonic stages, no differences can be detected between the youngest and the most advanced stages. Also the quantity of cementing substance and its extraordinary richness in glycoprotein are characteristic of the cornea."

Something similar holds for the reticular tissue of the spleen, etc. The reticular fibers of these organs develop, indeed, further than do the fibrils of the cornea, but never reach the stage of complete development of the collagen fibers, remaining on the contrary in an intermediate stage of development. They still retain a considerable amount of cementing substance, and the fibrils are relatively thin. The manner in which these fibers are stained with silver corresponds neither to embryonic nor to fully developed collagen fibrils.

The recognition of reticulin as a form of connective tissue whose development stops at an earlier stage than that attained by collagen fibers makes it impossible to refer to reticulin as a "precursor of collagen" or "procollagen", for fully developed reticulin never becomes collagen. However, it would be perfectly correct to say that collagen in its embryonic development passes through a stage corresponding to reticulin.

ANASTOMOTIC BRANCHING AS AN IDENTIFICATION MARK OF "RETICULIN" IN THE ANIMAL SKIN

In a recently published investigation on the morphology of collagen fiber bundles, Dempsey, Haines, and Hitchborn (7) have again tried to salvage Kaye's reticulin hypothesis as applied to the loose sheath tissue within the

corium. First they tried to differentiate the sheath tissue from the true collagen by means of various methods of silver impregnation staining. However they did not succeed in making the fibrils that surround and enclose the collagen fiber bundles stand out, using the silver impregnation procedure of Foot (8), which they consider to be most suitable and which stains reticular tissue black. They were equally unsuccessful in trying to stain these fibrils with specific elastin stains, a result reported by Seymour Jones more than 30 years ago (11). Nevertheless, these authors stick to the idea that this sheath tissue is a reticular tissue on the ground that its morphology resembles that of reticulin more than collagen. The critical difference is said to be the branching which is never observed in true collagen.

To establish that this sheath tissue shows real branching, these authors exhibit two photomicrographs of swollen collagen fibers, in which the constricting sheath tissue shows a somewhat doubtfully recognizable networklike branching, which however could just as well have been produced by networklike connections of unbranched individual fibers.

The question whether connective tissue structures show "true" branching of its fibers was studied some years ago by von Herrath and Dettmer (29), who carried out an electron microscope study of the reticulin of cat spleen. If true fiber branching is to be found in any connective tissue, surely it will be found in the genuine reticulin of those organs that gave rise to the term "reticular tissue" in the first place, because of the marked networklike branching. The electron microscope researches of von Herrath and Dettmer provide no evidence of a true branching of the reticulin fibers; rather, these consist, as in the case of collagen, of single, unbranched fibrils. The branching that is observed under the ordinary microscope is caused by a splitting of the bundles of fibrils into part-bundles, analogous to the splitting of collagen fiber bundles in the corium. The expression "reticular layer", applied to the part of the corium where the fiber bundle system is most thickly bedded and three-dimensionally ramified, recalls linguistically the expression "reticular tissue", both coming from the Latin *reticulum* (a net).

The same thing holds for the similar fiber branching, observed by means of the light microscope in the case of the elastic tissue. Just as in the case of typical reticulin, no true branching, having its origin in a branching within the molecule of the polypeptide chains, has been established. Here too the electron microscope shows unequivocally that the observed branching is produced by splitting of the fibril bundles. An example of this can be found in Figure 26, in the chapter on elastin in Neurath and Bailey's *The Proteins*, Vol. II, Part B, (1954).

Thus the impression of true branching, obtained from observations with the light microscope, has been shown to be erroneous by electron microscope studies in the cases of reticulin and elastin. It follows that visual microscopical observation of apparent branching, made by Dempsey *et al.* in the

case of the enclosing fibrils of the collagen fiber bundles of the animal corium, cannot be accepted as proof for true branching (anastomosis).

COLLAGEN STRUCTURES THAT DEVIATE MORPHOLOGICALLY

The collagen fiber system is quite variable in its morphological appearance and adapts itself to the existing biological environment. Examples are the abnormal growths arising from healing of wounds, including the overgrowth of brands and the healing of warble grub holes. The ossified parts of the skins of reptiles, etc., depart entirely from the usual collagen weave, not simply because of deposition of calcium compounds but also in respect to fiber weave pattern.

This holds true to an even greater extent for the extremely complicated structure of the collagenous ground substance in the true bones of vertebrates (30). Therefore it is not admissable that collagenous structures should beassigned, simply because of morphological variations, to a different connective tissue type, which was distinguished from collagen in the first place by histochemical peculiarities.

As far as concerns the loose fiber sheath tissue of the collagen fiber weave, this is to be grouped, morphologically, with the intramuscular connective tissue of the muscle strands. The latter, however, has always been considered to be collagenous in nature. The same holds for the collagenous portion of the neck ligament, the rest of which consists of elastin. This collagenous fraction, amounting to about 20% according to Bowes and Kenten, likewise forms an extremely fine fibrillar intermediate tissue that lies between the moderately thick elastin fibers and separates them from each other.

It is not apparent, therefore, why the fine fibrillar tissue between the fibers of the animal skin should be assigned to the reticular tissue type merely on morphological grounds. For the sake of an unambiguous nomenclature the word "reticulin" definitely ought to disappear from the vocabulary of tannery histologists and chemists.

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[In the 17th century] The trade in colonial leather and leather goods also extended along the entire coast. The exchange of leather was reciprocal, Pennsylvania supplying the Carolinas and in turn receiving from them. Both North and South Carolina leather found a market in New England, and the New Jersey tanneries sent their product to New York. Manufactures of leather were chiefly in the North, which also supplied Canada with this product. Massachusetts sent saddlery to the South and from the middle of the 17th century shipped shoes to the other provinces. There are records of consignments of Massachusetts shoes to New York and Philadelphia, and of exports from New Haven to the neighboring colonies. In 1769 a "Jersey shoe warehouse" at Albany traded largely in Newark manufactures. Meantime Pennsylvania itself supplied part of the southern trade, but largely as a distributor. In 1768 the recorded exports of Massachusetts were nearly 9000 pairs, while Pennsylvania imported 7918 pairs of American manufacture. The American shoes entered at the ports of all the Continental colonies, including Nova Scotia, were nearly 13,000 pairs.

—*History of Manufactures in the United States*, Vol. I, 1607–1860. Victor S. Clark. New York: McGraw-Hill Book Company, Inc., 1929.

REPORT OF WARING BLENDOR SUBCOMMITTEE
PREPARATION OF SOLID EXTRACTS FOR ANALYSIS

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F. LUVISI, AND R. L. YOUNG, JR.
with assistance of
R. M. LOLLAR

ABSTRACT

An interlaboratory experiment has shown that the proposed Waring Blendor technique for the preparation of solid extracts for analysis can be substituted for the present Official Method without significant influence upon the analytical values secured for the tannin analyses of the extracts. However, the statistically designed experiment by which this comparison was made has also yielded the additional information that the precision of the methods of analysis is not so good as is indicated in the present descriptions of the methods.

It is the Subcommittee's recommendation that the Tannin Analysis Committee adopt the Waring Blendor with the screen attachment as the Official Method for the preparation of solid extracts; further, that the Committee should consider experiments to find the causes of the low precision, and that particular attention be given to experiments to determine the between-day sources of variation in the analyses.

—x—x—

INTRODUCTION

At the June, 1956, meeting of the Tannin Analysis Committee Wagoner proposed a new method for the preparation of solid extracts for analysis. This method employed a Waring Blendor with special screen attachment instead of a mortar and pestle for grinding the portions of the sample making up the specimen used for analysis (Method A11, Par. 4.1.1). Data were presented to substantiate the method, and it was proposed that it be considered for possible adoption as an Official Method of ALCA. In keeping with the Association bylaws a subcommittee was appointed to study the proposal further. Inasmuch as there were to be five collaborating laboratories and four different extracts involved, it was decided to carry out the study on a statistical basis in order not only to evaluate the preparation method better but also to obtain comparative data on within-day, between-

*Publication of this report has been approved by a majority of the Tannin Analysis Committee, E. Lorne Rayfield, *Chairman*.

day, and between-laboratory reproducibility of both methods. Therefore, the assistance of Dr. R. M. Lollar was sought and obtained to set up and evaluate statistically the committee work.

OUTLINE OF COLLABORATIVE WORK

An experimental plan was drawn up and followed that would give (*a*) an unbiased comparison of the results secured from the Official and the proposed methods of preparation for four extracts by the five collaborating laboratories and (*b*) significant data on the within-day, between-day, and between-laboratory precision of the tannin analysis methods.

Twenty samples of each extract were prepared for analysis. Four samples of each extract were then selected at random and submitted to the respective laboratories. The cooperating laboratories made their analyses by both methods of preparation on four separate days. This was necessary since the maximum number of solutions that could be run on one day was eight. Two analytical solutions were made up from each preparation, and single determinations were made for total solids, soluble solids, and nontannins from each solution. The same analyses were repeated on another day using the duplicate sample. Thus, within-day and between-day results were obtained from each laboratory in addition to the between-laboratory results.

The cooperating laboratories were instructed to prepare the samples for analysis following their interpretations of Official Method A11, and by the use of the Waring Blendor with screen attachment as described in *JALCA*, **51**, 458 (1956). To be sure that all laboratories used the same-size screen, each was provided with a sheet of metal screening with ⅛-inch openings.

RESULTS

Tables I to IV show the comparative results obtained by the five collaborating laboratories. Table V is a tabulation of the significant data regarding conditions that prevailed during testing. The statistical evaluation data prepared by Dr. Lollar are shown in Tables VI and VII.

SIGNIFICANCE OF THE EXPERIMENTAL RESULTS

It is obvious from inspection of the data in Tables I to IV that the two methods of preparation of the extract for analysis have been without significant effect upon the results secured. This conclusion is supported by the statistical analyses of the data which are presented in Table VI, since the "Method" source of variance is not significant in any of the sixteen subtables. Since this source of variance is that due to the method of the preparation of the extract for analysis, i.e., the comparison of the present Official Method with the proposed Waring method, one can conclude that the Waring method could be substituted for the Official Method without influence on the analytical results to be obtained.

TABLE I

SUMMARY OF RESULTS OBTAINED BY FIVE COLLABORATING LABORATORIES ON *QUEBRACHO* EXTRACT

(Each result is the average of duplicate determinations)

Day	Preparation Time Seconds			Total Solids %			Soluble Solids %			Nontannin %			Tannin %		
	Off.	War.	Diff.	Off.	War.	Diff.	Off.	War.	Diff.	Off.	War.	Diff.	Off.	War.	Diff.
Elkland Leather Co.															
1	60	20	− 40	84.55	83.68	−.87	76.16	75.86	−.30	6.22	6.45	+.23	69.94	69.41	−.53
2	75	20	− 55	84.32	83.73	−.59	76.69	76.16	−.53	6.15	5.75	−.40	70.54	70.41	−.13
Diff.				.23	.05		.53	.30		.07	.70		.60	1.00	
U. S. Testing Co.															
1	48	19	− 29	83.88	83.95	+.07	75.67	75.65	−.02	7.42	7.38	−.04	68.25	68.27	+.02
2	34	9	− 25	84.47	84.52	+.05	75.89	76.15	+.26	7.36	7.31	−.05	68.53	68.84	+.31
Diff.				.59	.57		.22	.50		.06	.07		.28	.57	
Eastern Utilization Research Lab.															
1	120	13	−107	83.30	83.09	−.21	76.81	76.50	−.31	6.35	6.66	+.31	70.46	69.84	−.62
2	90	11	− 79	83.32	83.37	+.05	77.10	76.72	−.38	6.35	6.25	−.10	70.75	70.47	−.28
Diff.				.02	.28		.29	.22		.00	.41		.29	.63	
International Shoe Co.															
1	114	13	−101	82.67	82.83	+.16	75.13	75.17	+.04	7.26	7.35	+.09	67.87	67.82	−.05
2	180	10	−170	85.04	85.21	+.17	76.92	75.88	−1.04	6.93	7.17	+.24	69.99	68.71	−1.28
Diff.				2.37	2.38		1.79	.71		.33	.18		2.12	.89	
Armour Leather Co.															
1	83	14	− 69	82.48	82.40	−.08	74.81	74.92	+.11	6.01	5.92	−.09	68.80	69.00	+.20
2	57	10	− 47	82.78	82.73	−.05	75.40	75.28	−.12	6.03	5.99	−.04	69.37	69.29	−.08
Diff.				.30	.33		.59	.36		.02	.07		.57	.29	

TABLE II

SUMMARY OF RESULTS OBTAINED BY FIVE COLLABORATING LABORATORIES ON *WATTLE* EXTRACT

(Each result is the average of duplicate determinations)

Day	Preparation Time Seconds			Total Solids %			Soluble Solids %			Nontannin %			Tannin %		
	Off.	War.	Diff.	Off.	War.	Diff.	Off.	War.	Diff.	Off.	War.	Diff.	Off.	War.	Diff.
Elkland Leather Co.															
1	150	20	−130	84.15	84.04	−.11	82.38	82.76	+.38	17.14	17.37	+.23	65.24	65.39	+.15
2	165	35	−130	85.05	84.69	−.36	82.81	83.25	+.44	17.71	17.51	−.20	65.10	65.74	+.64
Diff.				.90	.65		.43	.49		.57	.14		.14	.35	
U. S. Testing Co.															
1	55	13	− 42	84.73	84.47	−.26	83.01	83.04	+.03	18.90	18.77	−.13	64.11	64.27	+.16
2	56	11	− 45	84.67	84.89	+.22	83.01	83.22	+.21	18.34	18.92	+.58	64.67	64.30	−.37
Diff.				.06	.42		.00	.18		.56	.22		.56	.03	
Eastern Utilization Research Lab.															
1	240	17	−223	83.88	84.08	+.20	82.65	82.65	.00	17.98	17.92	−.06	64.67	64.73	+.06
2	195	16	−179	84.31	83.81	−.50	82.70	82.25	−.45	17.86	17.55	−.31	64.84	64.70	−.14
Diff.				.43	.27		.05	.40		.12	.37		.17	.03	
International Shoe Co.															
1	118	13	−105	83.53	83.81	+.28	81.92	82.23	+.31	17.53	18.11	+.58	64.39	64.12	−.27
2	180	15	−165	85.50	85.00	−.50	83.12	83.38	+.26	18.32	18.09	−.23	64.80	65.29	+.49
Diff.				1.97	1.19		1.20	1.15		.79	.02		.41	1.17	
Armour Leather Co.															
1	170	13	−157	82.78	83.33	+.55	81.37	81.89	+.52	17.02	17.09	+.07	64.35	64.80	+.45
2	111	20	− 91	83.79	83.73	−.06	82.46	82.17	−.29	17.39	17.18	−.21	65.07	64.99	−.08
Diff.				1.01	.40		1.09	.28		.37	.09		.72	.19	

TABLE III

SUMMARY OF RESULTS OBTAINED BY FIVE COLLABORATING LABORATORIES ON *CUTCH* EXTRACT

(Each result is the average of duplicate determinations)

Day	Preparation Time Seconds			Total Solids %			Soluble Solids %			Nontannin %			Tannin %		
	Off.	War.	Diff.	Off.	War.	Diff.	Off.	War.	Diff.	Off.	War.	Diff.	Off.	War.	Diff.
Elkland Leather Co.															
1	180	30	−150	78.20	78.30	+.10	77.14	77.00	−.14	16.51	16.85	+.34	60.63	60.15	−.48
2	120	30	− 90	78.77	78.19	−.58	77.21	76.67	−.54	16.89	17.28	+.39	60.32	59.39	−.93
Diff.				.57	.11		.07	.33		.38	.43		.31	.76	
U. S. Testing Co.															
1	45	20	− 25	79.32	79.29	−.03	77.49	77.53	+.04	18.51	18.44	−.07	58.98	59.09	+.11
2	83	11	− 72	79.33	79.03	−.30	77.26	77.60	+.34	17.98	18.16	+.18	59.28	59.44	+.16
Diff.				.01	.26		.23	.07		.53	.28		.30	.35	
Eastern Utilization Research Lab.															
1	240	22	−218	78.37	77.94	−.43	76.74	76.45	−.29	17.40	17.20	−.20	59.34	59.25	−.09
2	210	26	−184	78.16	77.91	−.25	76.60	76.55	−.05	17.08	17.24	+.16	59.52	59.31	−.21
Diff.				.21	.03		.14	.10		.32	.04		.18	.06	
International Shoe Co.															
1	420	32	−388	78.86	78.75	−.11	77.36	77.00	−.36	17.88	17.67	−.21	59.48	59.33	−.15
2	480	30	−450	80.14	79.43	−.71	77.90	77.22	−.68	17.97	17.67	−.30	59.93	59.55	−.38
Diff.				1.28	.68		.54	.22		.09	.00		.45	.22	
Armour Leather Co.															
1	140	28	−112	77.99	77.69	−.30	76.48	76.24	−.24	17.02	16.88	−.14	59.46	59.36	−.10
2	143	36	−107	77.26	77.62	+.36	75.82	76.34	+.52	16.60	16.85	+.25	59.22	59.49	+.27
Diff.				.73	.07		.66	.10		.42	.03		.24	.13	

TABLE IV

SUMMARY OF RESULTS OBTAINED BY FIVE COLLABORATING LABORATORIES ON *SOLID CHESTNUT* EXTRACT

(Each result is the average of duplicate determinations)

Day	Preparation Time Seconds			Total Solids %			Soluble Solids %			Nontannin %			Tannin %		
	Off.	War.	Diff.	Off.	War.	Diff.	Off.	War.	Diff.	Off.	War.	Diff.	Off.	War.	Diff.
Elkland Leather Co.															
1	90	35	− 55	83.20	82.67	− .53	81.42	81.16	− .26	15.09	15.55	+ .46	66.33	65.61	− .72
2	110	25	− 85	82.41	82.89	+ .48	81.21	81.09	− .12	15.72	15.76	+ .04	65.49	65.33	− .16
Diff.				.79	.22		.21	.07		.63	.21		.84	.28	
U. S. Testing Co.															
1	46	13	− 33	82.93	83.54	+ .61	81.49	82.38	+ .89	16.83	17.15	+ .32	64.66	65.23	+ .57
2	45	11	− 34	83.35	82.28	−1.07	81.95	80.79	−1.16	16.87	16.71	− .16	65.08	64.08	−1.00
Diff.				.42	1.26		.46	1.59		.04	.44		.42	1.15	
Eastern Utilization Research Lab.															
1	150	14	−136	81.35	81.30	− .05	80.15	79.98	− .17	15.37	15.51	+ .14	64.78	64.47	− .31
2	120	12	−108	81.66	81.66	.00	80.33	80.44	+ .11	15.17	15.58	+ .41	65.16	64.86	− .30
Diff.				.31	.36		.18	.46		.20	.07		.38	.39	
International Shoe Co.															
1	300	15	−285	83.16	82.85	− .31	81.58	81.31	− .27	15.70	15.46	− .24	65.88	65.85	− .03
2	300	30	−270	83.77	83.27	− .50	81.69	81.54	− .15	16.84	15.62	−1.22	64.85	65.92	+1.07
Diff.				.61	.42		.11	.23		1.14	.16		1.03	.07	
Armour Leather Co.															
1	125	14	−111	80.89	81.62	+ .73	79.69	80.42	+ .73	15.06	15.24	+ .18	64.63	65.18	+ .55
2	90	13	− 77	81.60	81.58	− .02	80.47	80.30	− .17	15.44	15.32	− .12	65.03	64.98	− .05
Diff.				.71	.04		.78	.12		.38	.08		.40	.20	

TABLE V

TABULATION OF SIGNIFICANT DATA ON CONDITIONS DURING TESTING

Laboratory	Test	Soln. Temp. °C.	R. H. %	Barom. Reading in.	Oven Temp. °C.	Wet H. P. g.	Type Oven	Soln. Temp. °C.	R. H. %	Barom. Reading in.	Oven Temp. °C.
		QUEBRACHO						WATTLE			
Elkland	1	28	45	——	99	46.5	Conv.	28	45	——	99
	2	28	40	——	99	46.5	"	28	40	——	99
U. S. Testing	1	24.5	28	——	—	46.5	"	24.5	28	——	—
	2	24.0	30	——	—	46.5	"	23.9	30	——	—
East. Util.	1	23.5	46	29.83	100	46.0	"	23.5	46	29.83	100
	2	24.0	45	30.08	100	46.0	"	24.0	40	30.43	100
International	1	26.5	53	29.20	100	46.5	"	——	53	——	100
	2	23.5	84	——	100	46.5	"	23.5	77	——	100
Armour	1	24.5	31	29.78	99.5	46.3	Steam	24.5	31	29.78	99.5
	2	25.0	38	29.40	99	46.3	"	23.5	31	29.66	99
		CUTCH						CHESTNUT			
Elkland	1	27	39	——	99	46.5	Conv.	27	39	——	99
	2	28	40	——	99	46.5	"	28	40	——	99
U. S. Testing	1	24.5	32	——	—	46.5	"	24.5	32	——	100
	2	23.5	50	——	—	46.5	"	23.5	50	——	100
East. Util.	1	24.0	41	29.98	100	46.0	"	24.0	41	29.98	100
	2	24.0	45	30.08	100	46.0	"	24.0	40	30.43	100
International	1	25.0	69	29.99	100	46.5	"	25.5	69	29.99	100
	2	23.5	84	——	100	46.5	"	25.0	63	——	100
Armour	1	24.0	46	29.36	99.5	46.3	Steam	24.0	46	29.36	99.5
	2	25.0	38	29.40	99	46.3	"	23.5	31	29.66	99

TABLE VI

ANALYSIS OF VARIANCE TABLES

(Comparison of Results Obtained from the Waring Blendor and Official Techniques for the Reduction of Extract Size)

Source of Variance	Degrees of Freedom	Mean Square and Significance for Quebracho Extract	Wattle Extract	Chestnut Extract	Cutch Extract
Total Solids Analyses					
Method	1	0.1058	0.0280	0.0423	0.5107
Laboratory	4	3.6955	2.0992	6.1310**	4.2118**
M × L	4	0.2020	0.0672	0.1618	0.0572
Daywise replications	10	1.2385**	0.8130**	0.3702**	0.3094**
Within-day	20	0.0268	0.0256	0.0142	0.0221
Total	39	—	—	—	—
Soluble Solids Analyses					
Method	1	0.5244	0.2017	0.0325	0.2002
Laboratory	4	3.0327**	1.3133	3.9316**	2.2345**
M × L	4	0.1700	0.1151	0.0818	0.1845
Daywise replications	10	0.5000**	0.4630**	0.3742**	0.0971*
Within-day	20	0.0658	0.0513	0.0471	0.0305
Total	39	—	—	—	—
Nontannin Analyses					
Method	1	0.0022	0.0093	0.0032	0.0156
Laboratory	4	3.0941**	2.8694**	3.4152**	3.0596**
M × L	4	0.0251	0.0578	0.3366	0.0969
Daywise replications	10	0.0823	0.1606*	0.2159**	0.0969**
Within-day	20	0.0473	0.0531	0.0156	0.0272
Total	39	—	—	—	—
Tannin Analyses					
Method	1	0.5978	0.1243	0.0192	0.3240
Laboratory	4	5.9708**	1.1198*	1.6104*	1.0334**
M × L	4	0.2413	0.0791	0.3150	0.2222
Daywise replications	10	0.7985**	0.2550**	0.3861**	0.1231**
Within-day	20	0.0806	0.0582	0.0837	0.0369
Total	39	—	—	—	—

Daywise replications tested on within-day mean square; other factors tested on daywise-replication mean square as error estimate.

*Significant at the 5% level.
**Significant at the 1% level.

TABLE VII

SOME ESTIMATES OF THE PRECISION OF EXTRACT ANALYSES

Analysis	Extract	Maximum Value	Minimum Value	Grand Average	Standard Deviation		Precision Estimate in Present Official Methods
					Within-Day	Daywise Replication	
Total Solids	Quebracho	85.12	82.40	83.62	0.164	1.12	Duplicates are considered to be in good agreement when per cent total solids differs by no more than 0.2.
	Wattle	85.25	83.05	84.21	0.160	0.902	
	Chestnut	83.52	81.32	82.40	0.120	0.609	
	Cutch	79.79	77.44	78.53	0.149	0.556	
Soluble Solids	Quebracho	76.91	74.86	75.94	0.256	0.710	Duplicates are considered to be in good agreement when per cent soluble solids differs by no more than 0.2.
	Wattle	83.25	81.63	82.61	0.226	0.680	
	Chestnut	81.94	80.05	80.97	0.217	0.612	
	Cutch	77.56	76.08	76.93	0.175	0.312	
Non-tannins	Quebracho	7.40	5.95	6.61	0.217	0.287	Duplicates are considered to be in good agreement when per cent nontannins differs by no more than 0.2.
	Wattle	18.83	17.05	17.83	0.230	0.401	
	Chestnut	16.99	15.15	15.77	0.125	0.465	
	Cutch	18.47	16.67	17.40	0.165	0.311	
Tannin	Quebracho	70.61	67.84	69.33	0.284	0.894	Duplicates are considered to be in good agreement when per cent tannin differs by no more than 0.3.
	Wattle	65.42	64.19	64.78	0.241	0.505	
	Chestnut	65.97	64.58	65.17	0.289	0.621	
	Cutch	60.39	59.04	59.53	0.192	0.351	

It is also obvious from inspection of the data given in Tables I to IV that there are some rather large variations in the analytical results secured, which are ascribable both to the laboratories carrying out the analyses and to between-day sources of variation in the same laboratories. Table VI shows that both the laboratory effect and the daywise effect are generally significant. This is true both in the analyses for total solids, where laboratory manipulations are a minimum, and for nontannins, where more laboratory manipulations are involved. This fact indicates that the cause of this variation should be found in some factor common to all three of the primary analyses (total solids, soluble solids, and nontannins). This, therefore, suggests consideration of such factors as weighing and desiccator practice, dryer efficiency, and ambient atmospheric conditions such as relative humidity and air pressure.

It is noted that the one laboratory which employs the Alsop dryer and had generally low ambient relative humidities reported low total solids values. It may be that this reflects a lower degree of oxidation of the extracts due to

lower air flow in this dryer, coupled with better drying and retention of dryness during desiccation and weighing due to lower ambient humidity. The results were examined to see whether they indicated any dependence of total solids on ambient relative humidities; it was found that humidity values of 63% or greater did tend to yield high solids. Although it is not possible to make the positive statement that the ambient relative humidity has contributed to the variability shown in the present data, the results at least suggest that this is true.

The precision estimates contained in the present Official methods are not stated in terms of statistical parameters, but it is possible to deduce from the data given in Table VII that the methods are probably not quite as precise as the methods outlines would imply. The difference between two measurements, or their range, would be expected on the average to be 1.13 times the standard deviation. Table VII, therefore, shows that the within-day precision of results reported here is less than that claimed for the methods, to a slight extent, for all determinations except total solids. Based on the results secured in this work, it is suggested that ranges for duplicates of 0.20 for total solids, 0.25 for soluble solids and nontannins, and 0.35 for tannin might be anticipated. This precision estimate is rather meaningless, however, in view of the much larger and significant daywise replication standard deviation. Since all interpretation of analyses secured from the methods must involve comparison of results obtained on different days or in different laboratories (which inevitably involves different-day effects), the true precision estimate of the method should be the standard deviation estimates contained in the daywise replications. This fact indicates that further consideration should be given to the method to see whether the cause of the daywise effect can be determined in order that the effective precision of the methods may be improved. In this regard it is interesting to note that the daywise precision is lowest for the total solids, which involves the least laboratory manipulation. Thus, the importance of examination of the factors common to all three primary determinations such as weighing and desiccator practice or dryer efficiency, and their relationship to ambient atmospheric conditions, is again emphasized.

RECOMMENDATIONS

On the basis of these results the Subcommittee recommends that

1. The use of the Waring Blendor with screen attachment be adopted as the Official Method for the preparation of solid extracts.
2. The Tannin Analysis Committee reconsider the present "allowable difference limits in analysis" (J10) for tannin analysis in the light of the statistical evidence presented on the precision of

the methods. The source of the daywise variation should be studied in order that the precision of the methods may be improved.

ACKNOWLEDGMENT

The analytical work was carried out at the respective laboratories by the following individuals whose assistance is herewith gratefully acknowledged: G. Kelts and A. Volante, Jr. (Elkland Leather Company); H. J Willard (Eastern Utilization Research and Development Division of U. S. Department of Agriculture); J. K. Ghoee, A. Kramer, and E. Hill (International Shoe Company); A. Riner, B. A. Smith, and M. Neyhart (Armour Leather Company).

The chairman also acknowledges the assistance of W. G. Whitaker who prepared the samples and compiled the analytical data. Mrs. Margaret Ann Crawford performed the statistical calculations in the Tanners' Council Research Laboratory.

Received February 10, 1958.

CATTLE GRUB ERADICATION

The development of systemic insecticides or "grubicides" presents a challenging opportunity for the livestock, meat, and leather industries to embark on a program designated to eradicate one of the most damaging parasites affecting cattle and beef production—the cattle grub or warble. A brief description is given of how the grub affects the livestock, meat, and leather industries and the past attempt at cattle grub control.

The introduction of systemic chemicals provides a new concept of parasite control. The first systemic grubicide to receive approval for wide use is Dow ET-57. This product is not entirely without danger as concerns toxicity to livestock as its residues are found in milk and meat of animals for a period of at least 30 days following treatment. This precludes its use on dairy cows and on animals which are to be slaughtered for food purpose within 60 days following treatment.

The problem of toxicosis in man from pesticides has received continuing attention in medical circles. Pesticides are being used in the U.S. in about 80,000 preparations with over 200 basic chemicals. Human deaths ascribed to accidental poisoning by pesticides have ranged from 151 in 1946 to 104 in 1952. In about 50% of these cases death has been ascribed to arsenic compounds, but in from 10 to 20% the poison has remained unidentified.

Though the veterinary profession is well represented in the newly created National Cattle Grub Committee of Livestock Conservation, Inc., the question of how the veterinary practicioner fits into an organized effort to eradicate cattle grubs needs to be explored. The service that the individual veterinarian can render in this program will depend on (1) his knowledge of the drugs being employed, (2) his ability to differentiate between their side effects and manifestations of other diseases, and (3) his initiative in helping to solve the residue problem and in advancing a worthy project.

—From an editorial in *J. Am. Vet. Med. Assoc.*, **131**, 431 (1957).

LIFE LINES

Brief Biographies of Our Contributors

THOMAS J. CARTER, received the B. S. degree from Benedict College, Columbia, South Carolina, and has continued his education at Howard University and American University in Washington, D. C. He came to the National Bureau of Standards in 1943 and since that time has been active in the development of test methods and apparatus for the evaluation of leather and leather products. He became a member of ALCA in 1952.

DR. ADOLF KÜNTZEL has been a prolific contributor to the literature of leather chemistry since 1926. For more than 20 years he has directed the Institute of Leather Chemistry at the Technical Hochschule, Darmstadt, Germany. From this Institute there has come a steady stream of excellent papers dealing with almost every phase of the science of leather manufacture. Dr. Küntzel is a leading authority on the histology of skin. His massive chapter (175 pages) in the well known Bergmann-Grassmann "Handbuch der Gerbereichemie und Lederfabrikation" dealt with this subject with a thoroughness that no other treatise has even approached. In 1950 Dr. Küntzel founded the journal *Das Leder* as a successor to the old and respected *Collegium* and has served as its editor ever since. Dr. Küntzel not only consented to the republication of his paper on reticulin but also was kind enough to read the first draft of the translation, which benefited greatly by some of his suggestions.

JOHN F. WAGONER has been associated with Armour Leather Company, Williamsport, Pennsylvania for ten years and is now Technical Director of the Sole Leather Division. An active member of the Association, he served from 1939 to 1949 as Editorial Assistant of the *Journal* to which he now contributes as an abstracter.

NATIONAL CATTLE GRUB COMMITTEE COMPLETED

Dr. J. W. Cunkelman, *Chairman*, National Cattle Grub Committee, Livestock Conservation, Inc., has announced the completion of four committees to review and study grub control measures. The committees are: (1) Research and Evaluation, E. F. Knipling, *Chairman*, Harry Boyts, Dr. Fred O'Flaherty, John A. Killick, Dr. L. E. Swanson, and Jack Dreessen; (2) Economics of Grub Control, Roy Lilley, *Chairman*, Jim Rosse, Cecil Powell, L. E. Taylor, Earl McQuown, and Karl B. Musser; (3) Education and Information, C. E. Bell, *Chairman*, Doug Mossberg, Dr. C. M. Patterson, Blaine Liljienquist, and Frank Richards; (4) Program and Procedure, Herman Aaberg, *Chairman*, Ray Cuff, Dr. John Herrick, W. D. Knox, John Macfarlane, Dr. M. R. Clarkson, and Dr. E. E. Saulmon.
—*J. Am. Vet. Med. Assoc.*, **131**, 434 (1957).

ALCA NEWS

Proceedings, Reports, Notices, and News

of the

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DR. NAYUDAMMA CONFIRMED AS DIRECTOR

Dr. Y. Nayudamma, who has been Acting Director of the Central Leather Research Institute, Madras, India, since the death of Prof. B. M. Das, has been appointed Director of that Institute as of February 13, 1958. Dr. Nayudamma, a Mutual Member of the Association, is well known to many of our members, since he spent four years in the United States (1947–51) studying at Lehigh University under the late Dr. Theis. A brief biographical sketch of Dr. Nayudamma was published in *JALCA*, **52**, 94 (1957).

CONVENTION NEWS

An addition to the program of the Fifty-fourth Annual Meeting printed in last month's *Journal* has been announced by Joseph J. Stellbach, publicity chairman of the convention committee. A bus trip will be arranged from the New Ocean House, Swampscott, Mass., site of the meeting, to Lowell Technological Institute. The trip is planned to coincide with the Golf Tournament, which is scheduled for Monday afternoon, May 26, weather permitting.

Lowell Tech's Department of Leather Engineering, headed by Dr. Albert E. Chouinard, active ALCA member, was described in the May, 1957, *Journal*. The Department has complete research, testing, and processing laboratories for leather, including a pilot-plant tannery. It offers the only course in the country leading to the degree of B.S. or M.S. in Leather Engineering.

In the Lowell Technological Institute Research Foundation Dr. Thomas C. Thorstensen is conducting the Tanners' Council Beamhouse Research Project under the sponsorship of 27 companies. This project seeks to develop new beamhouse procedures with particular emphasis on chemical and enzymatic unhairing methods.

Other departments of the Institute will also be open to visitors, including paper, plastics, electronics, and textile areas. The last-named annually attracts hundreds of visitors from all over the world and will be of particular interest to the ladies.

ANNUAL REPORT OF ALCA TECHNICAL COMMITTEES

January 10, 1958

Methods revision and status.—At the meeting in January, 1957, it was brought to the Council's attention that the status of most of the technical methods of the Association had changed since the revised methods were published in 1955, and that many methods, particularly the B series, had undergone many changes in wording, procedure, and meaning. It was decided by Council

1. That a tabulation of the methods should be made showing the 1957 status of each method and whether or not the wording of the method had been changed.
2. That all changes and corrections made in each method would be printed on a separate sheet so it could be inserted in the methods book adjacent to the proper method.
3. That the whole B series should be reprinted.

This work is all done, the type has been set, the proofs corrected, and the methods are being printed.

Committee appointments.—Since the Council meeting in October, 1957, Dr. Wallace Windus has been appointed as chairman and Fred Luvisi has been added to the membership of the Hide Powder Committee.

Committee reports.—The annual report of the chairman of each technical committee is submitted as part of this report. At the present time all the committees are dealing with new methods or changes in old methods which require additional committee work before they are ready to be considered for adoption by the Association, and no Council action is required at this time on any committee work.

M. Maeser
President-Elect, ALCA

REPORT OF THE HIDE POWDER COMMITTEE

Wallace Windus, *Chairman*

G. Daub
G. V. Downing
A. N. Kay
F. P. Luvisi
F. F. Marshall
G. W. Schultz

The main activity of the above committee consisted in the analysis and approval of Lot 28 hide powder in comparison with Lot 27 during the fall of 1956. Mr. Marshall does not anticipate the need for another lot of hide powder until 1958.

Ira D. Clarke retired from the Hides, Tanning Materials and Leather Section of the Eastern Utilization Research and Development Division, United States Department of Agriculture, on October 15, 1957, and resigned the chairmanship of this committee. He has been succeeded as chairman by Dr. Wallace Windus. Fred P. Luvisi has been appointed as a new member.

REPORT OF THE INDUSTRIAL WASTE COMMITTEE

J. F. Wagoner, *Chairman*

C. A. Butz
Ben A. Crota, Jr.
W. C. Curtin
J. W. Harnly
M. M. Luckens
E. E. Presley
Ernest H. Stein
J. E. Tarman

Council agreed last year that this committee's activities should be confined to serving as a clearing house for information relative to stream pollution abatement. Therefore, the activities during the past year have been limited to answering a few inquiries on this subject.

REPORT OF THE MINERAL LEATHER COMMITTEE

N. C. Benrud, *Chairman*

R. Adams
F. O'Flaherty
T. S. Perrin
L. Seligsberger
R. Stubbings
T. Weaver

J. Wendkos

Mr. Adams resigned as chairman of this committee, and N. C. Benrud was appointed to fill his place. Mr. Adams agreed to continue as a working member.

The Committee has been working on the Lehigh basicity and chromic oxide determination.

A review of the work done by the committee during the last several years has been completed. Some of the data have been rechecked. The members of the committee have decided to have this material written up and presented for publication. Although the project is not completed, it will serve as a guide for anyone working independently on this basicity method.

Work will be continued on the Lehigh basicity method and a comparison made with the present ALCA method.

REPORT OF THE RAW STOCK EVALUATION COMMITTEE

Thomas C. Thorstensen, *Chairman*

M. Battles
J. Highberger
R. I. Miller
E. J. Strandine
R. Stubbings
C. D. Wilson

With the sudden loss of William Cox as chairman of the Raw Stock Evaluation Committee, Thomas C. Thorstensen was appointed as chairman. This committee met during the Association convention at Lake Placid.

The purpose of this committee is to develop practical methods of hide evaluation if the committee believes such can be done. If in the opinion of the committee such methods are not practical, that should also be reported.

Dr. Lollar discussed the determination of hydroxyproline and of soluble hydroxyproline as a method of hide evaluation and reported that as yet the method is not sufficiently accurate to be considered a possible means of raw stock evaluation.

Dr. Highberger remarked that the hydroxyproline method is probably a good method of characterizing soluble collagen but what is also important is the condition of the fiber which may or may not be measurable by such a method.

The committee then discussed the question of the effects of cure upon leather quality, possible methods of measuring cure, and the value of cure evaluation methods in the light of present hide marketing practice.

The chairman asked the opinion of the committee on the value of analysis methods for salt, ash, moisture, and manure. The committee voted against this approach also.

The chairman asked the committee whether in their opinion an attempt should be made to develop a practical cure index. The committee voted against this approach.

The committee then discussed possible approaches to raw stock evaluation. There was general agreement among the committee members that raw stock evaluation methods in order to be practical must be accurate and significant. There are no methods which are obviously of value in the solution of this problem.

REPORT OF THE PHYSICAL AND MECHANICAL PROPERTIES COMMITTEE

Ell Dee Compton, *Chairman*

With the approval of Council this committee was reorganized to consist essentially of the chairmen of subcommittees and sections of the ALCA-ASTM Joint Committee on Leather. The ALCA-ASTM Joint Committee is considering each of the physical test methods of ALCA for presentation as ASTM methods as well as ALCA methods. As a part of this work each of the ALCA methods is being reconsidered as such. In each instance the method will be presented in the standard form of the respective association. The ALCA Council has authorized the Physical and Mechanical Properties Committee to approve publication of new methods, or present ALCA methods in ASTM form, as joint provisional methods. For a procedure to become an Official Method of the ALCA—or the ASTM—it must be approved by the entire association.

The membership of the Physical and Mechanical Properties Committee and the responsibility of each member in the ALCA-ASTM Joint Committee are as follows:

	ALCA-ASTM		
Individual	Subcomm.	Section	Study Area
R. G. Ashcraft, *Chairman*	III	4	Measurement of Water Resistance
M. M. Baldwin, *Chairman*	IV	—	Research
G. A. Butz, *Chairman*	III	—	General Physical Testing
A. N. Compton, *Chairman*	III	7	Mechanical Leathers
E. D. Compton	III	5	Lightfastness of Leather
R. J. Kanagy, *Chairman*	Joint Committee		Water Resistance of Sole Leather
R. M. Lollar, *Chairman*	I	—	Nomenclature, Definitions, and Applicability
A. N. Kay, *Chairman*	III	10	Miscellaneous Physical Properties
C. W. Mann, *Chairman*	II	—	Sampling and Conditions for Physical Testing

Individual	ALCA-ASTM Subcomm.	Section	Study Area
J. Naghski, *Chairman*	III	2	Physical Dimensions
W. T. Roddy	—	—	———
J. S. Rogers	—	—	———
R. Stubbings, *Chairman*	III	5	Surface Characteristics
R. Young, *Chairman*	III	3	Tensile Properties

Almost all of the work done under the ALCA-ASTM Joint Committee on Leather is being done by members of ALCA. In the following summary of the methods under study ALCA members primarily responsible for the particular study are listed even though they may not be members of the Physical and Mechanical Properties Committee. In most of the studies being made, other ALCA members are quite active, but only the person primarily responsible for the study is listed.

ALCA Method No.	Subject	Status	Individual	ALCA-ASTM Sub-comm.	Sec.
E1	Conditioning Leather for Physical Testing	Draft being reviewed by Nomenclature Committee	C. W. Mann R. M. Lollar	II I	— —
E2	Area	Draft to Nomenclature Committee	J. Naghski R. M. Lollar	III I	2 —
E5	Width	Draft to Nomenclature Committee	J. Naghski R. M. Lollar	III I	2 —
E3	Thickness of Units	Under study	J. Naghski	III	2
E4	Thickness of Specimens	Under study	J. Naghski	III	2
E10	Tongue Tear Strength	Being drafted	R. Young	III	3
E11	Buckle Tear Strength	Being drafted	R. Young	III	3
E12	Stitch Tear Strength, Single Hole	Being drafted	R. Young	III	3
E13	Stitch Tear Strength, Double Hole	Being drafted	R. Young	III	3
E14	Bursting Strength, Ball	Being drafted	R. Young	III	3
E15	Tensile Strength	Being drafted	R. Young	III	3
E16	Grab (Break) Strength	Being drafted	R. Young	III	3

ALCA Method No.	Subject	Status	Individual	ALCA-ASTM Sub-comm.	Sec.
E17	Elongation	Being drafted	R. Young	III	3
E17A	Elongation—Alternate Method	Being drafted	R. Young	III	3
E31	Water Resistance (dynamic), Upper Leather	Being drafted*	R. G. Ashcraft	III	4
—	Water Resistance (dynamic), Garment	Under study	A. Chouinard	III	4
—	Water Resistance (dynamic), Sole	Under study	J. Kanagy	III	4
—	Water Resistance (dynamic), Glove	Under study	C. Mann	III	4
E30	Water Resistance (static), Upper	Under study	R. G. Ashcraft	III	4
E30	Water Resistance (static), Sole	Being drafted	R. G. Ashcraft	III	4
—	Water Resistance (static), Glove	Under study	A. Chouinard	III	4
—	Lightfastness of Leather	Under study†	E. D. Compton	III	5
E46	Crocking	Under study	P. McKee	III	5
E42	Resistance to Cracking at Low Temperature	Under study	P. McKee	III	5
—	Scuff Resistance	Under study	P. McKee	III	5
—	Flex Resistance	Under study	H. Haemiseggar	III	5
—	Deterioration of Insole Leather	Under study	S. Kreman	III	8
—	Bond Strength of Belting Leather	Under study	A. N. Compton	III	9
—	Corrosion of Metal in Contact with Leather	Under study	A. N. Compton	III	9
—	Test for Oil or Air Permeability of Leather Cup Packing	Under study	A. N. Compton	III	9
E43	Stiffness	Under study	M. Bailey	III	10
E40	Break	Under study	M. Bailey	III	10
E45	Compressibility	Out for Committee vote	A. N. Kay	III	10

*An extensive study was made, using seven methods, nine laboratories, three determinations each. The Maeser method is to be rewritten using an electrical end point and a higher speed.

†An extensive study utilizing nine leathers, five laboratory methods of test in eleven laboratories, and outdoor exposure at six periods of the year.

Dr. M. M. Baldwin, Battelle Memorial Institute, is chairman of Subcommittee IV on Research. He has requested that he be notified of any methods requiring research or of new methods needed. The purpose of his committee is to develop new methods which are needed in our field.

REPORT OF THE PREPARATORY AND POSTTANNING PROCESSES COMMITTEE

W. T. Roddy, *Chairman*

Fats and Oils Subcommittee

C. A. Blair, *Chairman*
B. A. Grota
V. Mattei
C. M. Morrison
C. E. Retzsch
T. Weaver

When the chairman accepted this post a few years ago, the work to be performed consisted of rewriting the F and G series for the Methods Booklet. As this appeared to be mainly a one-man job and as there was no working committee, the chairman accepted the task and rewrote the methods. The methods have been accepted by the membership.

At the time of accepting the chairmanship there was one active subcommittee under the jurisdiction of this committee, the subcommittee on Fats and Oils with R. M. Koppenhoefer as chairman working on the "Total Oil" Method listed in the H series. When Dr. Koppenhoefer resigned, his position was taken by Mr. Leach, a member of the subcommittee recommended by Dr. Koppenhoefer and accepted by W. T. Roddy upon approval of Council. The only active work being performed at present is by the subcommittee.

As agreed upon at the meeting of the subcommittee on June 4, 1957, at the Lake Placid Club, a proposed method of analysis for free and combined oil in leather was sent by Mr. Leach to each committee member on July 19. Criticism and comments on the method were requested.

Upon receipt of returns from the several members a modified method will be written up based on the opinion of the majority of the subcommittee members. This modified method will then be mailed to each member for use in analysis of the sample of chrone retan leather that each member has on hand to be tested. The subcommittee's work on the method is necessary because of the rather widespread differences in the free and combined oil analyses on a previous sample which definitely pointed to a need for a more accurate method of analysis that will permit better correlation between laboratories.

When the proposed method has been modified to the satisfaction of all members, the analyses will be performed and the results sent to Mr. Leach for examination and compilation. In keeping with the request made by M. Maeser, President-Elect, Mr. Leach has been informed that a subcommittee report is not necessary until the work has been completed.

REPORT OF THE STANDARDS AND SPECIFICATIONS COMMITTEE

Carl G. Telander, *Chairman*

R. G. Ashcraft
M. H. Battles
S. Dahl
B. A. Grota
A. N. Kay
K. H. Knight
R. M. Lollar
C. W. Mann
E. E. Presley
R. L. Young, Jr.

The following matters pertaining to Federal standards and specifications have been presented for consideration of the committee:

The question of sampling of sole leather on an area vs. weight basis has been studied by the committee in detail. It has been conclusively shown that there is no significant difference by either method, and the committee therefore recommended that for the sake of simplicity and to minimize sources of error the sampling be done with a die. The report will be presented under the authorship of C. W. Mann to the Editor of the *Journal*.

The question of moisture and grease determination (chloroform vs. petrolic ether) and its influence on the degree of tannage, etc., has been discussed. It has been pointed out that in some borderline cases a change of extractant gives differences which are sufficient to accept or reject a leather under the Federal specifications. The matter is of sufficient importance to require a separate study and has been referred to the Vegetable Leathers Committee.

The committee has made its recommendations to the Leather Section of the Bureau of Standards in regards to a new method for alkali solubility of leather-cellulose mixtures. Several matters of physical testing methods presented to the chairman were referred to the appropriate committee.

REPORT OF THE TANNIN ANALYSIS COMMITTEE

E. L. Rayfield, *Chairman*

R. E. Barnwell
M. Baum
T. C. Blair
J. H. Davis
E. Facer
J. Jany
E. P. Luvisi
F. F. Marshall
R. L. Moore
L. Sheard

G. W. Stanbery

Subcommittee on the Determination of Lignosulfonates

Sverre Dahl, *Chairman*

D. M. Balfour
R. Barnwell
J. Giguere
J. M. Hinkle

Virginia R. Power

Subcommittee on Waring Blendor

J. F. Wagoner, *Chairman*

E. Facer
A. N. Kay
F. Luvisi
E. L. Rayfield
R. L. Young, Jr.

Further tests have been carried out by this subcommittee on the preparation of solid tanning extracts for analysis.

The Subcommittee on the Determination of Lignosulfonates has had a report published this year on this subject: "A Collaborative Study on the Cinchonine Method for Determination of Lignosulfonates in Vegetable Extract Blends".

The same subcommittee has been making further investigations on different methods for the above.

One interesting method was translated into English from Japanese by Kenkichi Tomita, Enzyme Institute, University of Wisconsin, through the auspices of The Marathon Corporation and Martin Baum. It is entitled "The Selective Absorption of Lignosulfonic Acid and Vegetable Tannin by Hide Powder" and is being studied, but as yet there is nothing further to report.

REPORT OF THE VEGETABLE LEATHERS COMMITTEE

A. N. Kay, *Chairman*

R. G. Ashcraft
C. Blair
I. D. Clarke (retired)
S. Dahl
G. V. Downing
R. M. Lollar
F. F. Marshall
G. W. Schultz
J. F. Wagoner

Committee work, this year, has been the continuation of the studies of the methods of determination of moisture (B3) and solvent extract (B4) by a subcommittee under the chairmanship of C. Blair. This consists of I. D. Clarke, R. M. Lollar, S. Dahl, F. F. Marshall, J. F. Wagoner, and A. N. Kay, assisted by Drs. A. N. Compton and H. B. Merrill.

Because these two determinations have been sources of dissatisfaction and controversy ever since the founding of the Association, the subcommittee is hoping to be able to settle them once and for all. Under the circumstances, an early solution is hardly to be expected. Since a good deal of basic work is required, it was agreed that Mr. Blair (chairman), Dr. Lollar, and Mr. Kay should form a small task-force to look into these basic matters.

Moisture.—Because of the volatility of certain oils used in leather, a solvent-extraction method seems likely to be the most exact, and the task force is considering the different types of apparatus to find one sufficiently

precise. Under consideration, too, is what may justly be considered moisture, when leather itself is known to contain "free" and "bound" water and water of constitution, while many of its other components contain water of crystallization (e.g., epsom salts) or of constitution (tannins, etc.), part or all of which may, or should, be retained at usual temperatures and so be measured, again, in other parts of the analysis. As Dr. Lollar has shown, different solvents give different results.

Solvent extract.—Under this heading the task force has also made progress. The basic problem is to find a solvent which can be considered to remove substantially all the fatty material from the leather—but only that material. Dr. Lollar's careful studies have confirmed the earlier belief that chloroform extracts nonfatty material from "loaded" leathers (e.g., sole). A survey of some 180 solvents by Dr. A. N. Compton indicated that only a handful had promise for the purpose.

The work on both fronts is being continued, and it is recommended that Methods B3 and B4 be continued as Provisional.

In expressing his appreciation of the interest and encouragement of the whole committee and particularly of Drs. A. N. Compton and H. B. Merrill, the writer wishes to take special note of the tremendous contributions made by Mr. Blair and Dr. Lollar in this difficult and tedious work.

REPORT OF LEATHER FINISHES COMMITTEE

Bernard Roll, *Chairman*

A. G. Fleisch
C. P. McKee
R. V. Osborne
A. Phillips
E. Presley
R. Shaw
T. Weaver
R. Welch
C. D. Wilson

The committee has been responsible for Methods K1, K5, K10, and K11 which are now Official as well as for Method K12 which is now Provisional. No change in status or wording of these methods is indicated at the present time.

Messrs. Phillips, McKee, Presley, Wilson, Shaw, Welch, and Osborne have carried out experiments designed to measure quantitatively the propensity of finished leathers to "bleed" on to white stitching. Several interesting procedures yielding reasonably good results have come out of this work. The problem now is to try to select the best possible procedure and see it through to a conclusion. Mr. Osborne has prepared all the stitched, finished leather specimens used in this project.

Elwood Presley has offered a test procedure for measuring "bleeding" of finished leathers on to various types of fabrics. Early results have been very encouraging, and we hope to have enough data to warrant its proposal as a provisional method very soon.

Several publications have appeared in the British *Journal* during the past few years dealing with procedures for evaluating leather finishes. Unfortunately, only a few of our committee have access to this publication, but a great deal might be gained if these papers could be made available to all our committee members.

ABSTRACTS

Studies on Vegetable Tanning. II. Some Consideration of Particle-Size Distribution for Vegetable Tannins. Y. Inouye and H. Okamura. *Bull. Japanese Assoc. Leather Technol.*, **3**, 219–24 (1957) (English summary).—When wattle extract solution was precipitated by adding successively larger concentrations of salt, it was obvious that the tannins precipitated by addition of a small amount of NaCl had larger particle sizes than those precipitated at higher salt concentrations, but none of the precipitated fractions seemed to consist of particles of uniform size. An ion-exchange technique was employed to study the particle-size distribution, employing 3 polystyrene sulfonates (Na form) of different degrees of cross-linking (DVB 1.0, 5.0, and 10.0%). Although tannins, if ionized at all, would produce anions, it was found that the tannin was absorbed to some extent by cationic exchangers, and the amounts absorbed decreased with increasing degree of cross-linking of the exchanger. Takeup of tannin by the exchanger is believed to be a case of molecular absorption. From the difference in quantity of tannin absorbed by two different exchangers, the molecular size of the tannins can be deduced.

Studies on the Enzyme Depilation of Hides and Skins. II. Bacteriological Characteristics of Selected Strains. H. Toyoda and A. Futami. *Bull. Japanese Assoc. Leather Technol.*, **3**, 225–32 (1957) (English summary).—Strains previously found to produce very active depilatory enzymes were identified as *B. subtilis* (2 strains), *B. megatherium*, *B. pumilus*, and *B. cereus*.

Recovery of Tannin from Western Hemlock Bark. I. Extraction Rates with Water as a Solvent. D. S. Scott. *Pulp and Paper Mag. Can.*, **57**, 139–41 (1956).—Samples of hemlock bark taken from hydraulic debarkers were analyzed. Results indicate that tannin content declines to 8–9% and purity declines to 50–56 after the logs have been in fresh and salt water for 5–6 months. When samples of the bark were cooked with 10 parts of water for periods up to 3 hr., only 50% of the analytically determined tannin was extracted at 80°C. and only 70% at 100°. The extraction rate is given by the expression $W=kt^n$ where W is percentage of dry bark weight extracted, t is

temperature, n is independent of t, and k varies with t and with quality of the bark. Results of pilot-plant extractions agreed with laboratory-scale tests. **II. Extraction Rates with Dilute Alkaline Solvents.** D. S. Scott and P. Korach. *Ibid.*, 147–50.—Yields of tannin were greatly improved, and extraction hastened, by cooking the bark with 10 parts of solution containing enough alkali to give final pH values from 6.5 to 7.5, e.g., 2 lb. Na_2CO_3 or 1 lb. NaOH per 100 lb. bark. When extraction was done at 100°C., tannin recovery exceeded the analytically determined percentage, and purity was not materially affected by the small concentrations of alkali used. The resulting liquors were free from insoluble matter, but some precipitation occurred when pH was lowered to 5.0. Bubbling air through the liquor during the cook apparently did not impair the quality of the extract. The liquor was not attacked by molds. Fine grinding increased the rate of extraction, but not enough to justify the added cost; however, a grind fine enough to give a slurry that can be pumped (10 to 40 mesh) is advised. H.B.M.

Spectrophotometric Determination of Iron with Ethylenediamine Di(o-Hydroxyphenylacetic Acid). A. I. Underwood. *Anal. Chem.*, **30,** 44–47 (1958).—This reagent gives a red color through the formation of a very stable chelate with ferric iron. Maximum absorption is at about 4700 A; a blank on the reagent is required. Absorbance is independent of pH value from 2 to 9; determinations are made at about pH 5. A large excess of the reagent is unnecessary. Optimum iron concentration is 3 to 8 p.p.m. At 6 p.p.m. the standard deviation of the determination is 0.53%. Chromium interferes, but Al, Ca, Pb, and Mg do not. Copper does not interfere when present in a ratio of 7:1 to iron, but interferes at 70:1. H.B.M.

The Solubility and Properties of a Purified Ichthyocol in Salt Solutions of Neutral pH. P. M. Gallop, S. Seifter, and E. Meilman. *J. Biophys. Biochem. Cytol.*, **3,** 545–57 (1957); *Chem. Abstr.*, **52,** 470*h*.

The Correlation between Function and Chemical Composition of the Collagen Fiber. I. Banga. *Schweiz. med. Wochschr.*, **86,** 1045–47 (1956); *Chem. Abstr.*, **52,** 484*f*.

The Wettability of Chrome-tanned Leather. R. G. Mitton and C. D. Pomeroy. *J. Soc. Leather Trades' Chemists*, **41,** 402–17 (1957).—During the drying of chrome-tanned leather H_2SO_4 escapes in small quantities, and the process directly affects wettability. The resultant changes in chrome complex are probably associated with the changes of feel which occur during aging or intensive drying. Changes in chrome leather wettability resulting from drying were studied by varying chrome content, temperature of tanning, pH of neutralization, and temperature and duration of drying. As the temperature of drying increased beyond 40°C. the pH difference of water extracts of grain and adjacent layers increased. Water adsorption measurements suggest that the changes in the chrome complex that occur during leather aging take place faster at higher temperatures, and at temperatures near 80°C. the leather sur-

face becomes much more water-repellent than the rest of the leather. Water penetration pressures determined on chrome leather made from degreased pelt were raised by thorough neutralization, by increasing the amount of fat applied to the degreased and then tanned pelt, and by using stearic acid rather than oleic acid. J.M.C.

A Survey of Skin Diseases of Cattle in Kenya and Their Effect on the Finished Leather. H. F. Green. *J. Soc. Leather Trades' Chemists*, **41**, 418–23 (1957).—At least 25% of Kenya cattle hide production is affected by disease. The survey deals with identification of individual diseases, their prevalence, and their effect on hide structure and on finished leather. Diseases are described and illustrated as they appear on dry raw hides, in sections through lesions, and on finished leather. Demodicosis and streptothricosis are most prevalent, the former being the most damaging. Relatively uncommon are photosensitization, ringworm, and mange. J.M.C.

The Migration of Salts in Leather. R. G. Mitton. *J. Soc. Leather Trades' Chemists*, **41**, 424–30 (1957).—Experiments are described showing very little movement of salt in air-dry chrome or vegetable-tanned leather. Dipping one end of a vegetable-tanned leather in a salt solution results in rapid movement of salt and growth of salt crystals on the exposed leather. The movement of salt is apparently due to capillary flow of salt solution followed by evaporation. The migration of salt is very dependent on the nature of the vegetable tannin, on the presence or absence of oil in the leather, and on the availability of air. J.M.C.

The Conversion of Lysine to Hydroxylysine and Its Relation to the Biosynthesis of Collagen in Several Tissues of the Rat. K. A. Piez and R. C. Likens. *J. Biol. Chem.*, **229**, 101–9 (1957).—Collagen from skin, tail tendon, bone, and teeth (dentin) of rats given lysine — C^{14} was chromatographically analyzed for lysine and hydroxylysine content. The ratio of total lysine to hydroxylysine was 6.0 in skin, 3.9 in tail tendon, 1.9 in bone, and 1.1 in dentin. The presence of large amounts of exogenous hydroxylysine in the plasma did not alter the degree of conversion of lysine to hydroxylysine in any of the collagens. The results are interpreted as indicating that free hydroxylysine cannot be used for collagen synthesis, lysine providing the only source. The conversion apparently takes place in the bound form of lysine and may involve the hydroxylation of a collagen-like precursor originally containing no hydroxylysine. J.M.C.

The Interaction of Silicic Acid with Collagen and Gelatin Monolayers. S. G. Clark and P. F. Holt. *Trans. Faraday Soc.*, **53**, 1509–15 (1957).—The properties of monolayer films of tendon collagen solubilized in acetic acid, of hide powder collagen solubilized in formic acid, and of gelatin were examined by a study of surface pressure measured with a Langmuir-Adam balance using a waxed Perspex trough maintained at 25°C. The films of gelatin and acetic acid-soluble hide collagen behaved in an almost identical manner, both being

more compressible than films of globular proteins. The films of tendon collagen resemble the films of gelatin and hide powder collagen but show differences in detail, the differences being mainly due to the fact that films of tendon collagen were constructed from least-degraded material. In contrast to films of globular proteins the force required to bring a collagen or gelatin film to an arbitrary specific area showed very little variation with pH. When the solution beneath collagen or gelatin films contains silicic acid which has not been prepolymerized, the characteristics of the films are scarcely altered by the silicic acid. The interaction of prepolymerized silicic acid with collagen and gelatin is shown by increases in the film areas. With concentrations of silicic acid 0.001*M* or more the interaction occurs over a pH range 3 to 8, the same range as was found with albumin and insulin. Tanning of the films is shown by decreases in compressibilities, larger specific areas, and solidification of the films. Tanning of collagen films is probably due to cross-linking of chains rather than to polymerization of silicic acid beneath the film as is the case with globular proteins. J.M.C.

"Nonswelling" Acids. G. Otto. *Das Leder*, **8**, 100–6 (1957).—Acids that neither swell nor tan hide (discovered by M. Bergmann and coworkers) are of advantage for deliming and for pretanning treatments. They attach themselves to the hide so firmly that it can be washed hundreds of times before all the acid is washed out. When these acids are used, vegetable tanning is faster, distribution of the tannin is more uniform, the leather color is lighter, the grain is finer and more elastic, and yield and fullness of the leather are often better. The causes of swelling are discussed; swelling may be ionic (by H ion) or lyotropic. If the anion has an affinity for collagen, swelling of the skin is repressed. J. Steinhardt *et al.* (*J. Research Natl. Bur. Standards*, **30**, 123 [1943]) developed a method for determining the affinity of anions for fibrous proteins. The affinities for collagen of a few organic acids were determined by this method and were found to be about the same as their affinities for wool as reported by Steinhardt. These phenomena have been discussed previously (see abstracts, *JALCA*, **49**, 123, 642 [1954]). The behavior of acids having a specific anion affinity was studied by treating pieces of isoelectric calfskin with solutions of 16 different aromatic sulfonic acids and their neutral (pH 6.5) sodium salts. Only acids that had no tanning action were used; skin treated with any of these acids and dried was transparent. Portions of calfskin were shaken for 20 hr. at 25°C. with 2000% of their weight of 0.05*M* solutions of the acids or their salts. Swelling (S) was measured by first weighing the isoelectric skin after blotting it with filter paper, then reweighing after the acid or salt treatment, and expressing the increase in weight (S) in relation to that of the isoelectric skin taken as 100. Determinations also were made of shrinkage temperature (Ts) and dissolved skin decomposition products (soluble hide) by determining N_2 in the used solutions. The results are shown in the following table, which also gives data for water and 2 mineral acids.

The first 7 acids (Nos. 1 to 7) are single-ring acids with, in general, increasing possibility of dipolar activity. For acid No. 1 Ts is only silghtly higher than that for mineral acid, but with increasing dipolar activity S decreases and Ts increases to values approaching those for isoelectric skin. The double-ring acids (Nos. 8 to 14) all have higher dipole activity; they swell skin very little and lower Ts only a few degrees. The naphtholsulfonic acids (Nos. 10 and 12),

No.	Acid	Acid S	Acid Ts	Acid Soluble Skin %	Salt S	Salt Ts	Salt Soluble Skin %
–	water	—	—	—	100	66	—
–	hydrochloric	258	42	0.8	—	—	—
–	sulfuric	235	43	0.3	—	—	—
1	benzenesulfonic	198	49	0.2	100	65	—
2	*p*-phenolsulfonic	193	58	0.4	110	63	0.2
3	aminophenolsulfonic	164	60	0.3	107	64	0.1
4	*p*-dodecylbenzenesulfonic	169	49	0.7	148	48	4.2
5	*p*-nitrobenzenesulfonic	122	62	—	103	59	0.3
6	*p*-nitrobenzenedisulfonic	129	64	—	118	62	0.7
7	β-sulfophthalic	112	62	—	139	60	0.9
8	*p*-hydroxydiphenylsulfonic	107	62	—	105	60	—
9	β-naphthalenesulfonic	111	61	—	112	62	0.4
10	naphthol-3-sulfonic	75	63	—	142	59	1.4
11	naphthalene-2,5-disulfonic	125	60	0.2	150	57	0.7
12	naphthol-3,6-disulfonic	87	63	—	141	52	0.9
13	diisopropylnaphthalenesulfonic	112	59	0.8	134	47	2.5
14	diisobutylnaphthalenesulfonic	110	60	0.6	126	42	7.6
15	anthraquinone-β-sulfonic	70	68	—	102	65	0.3
16	polystyrolpolysulfonic	95	68	—	101	66	—

which can unite to peptide groups through H bonds, repress swelling, lower Ts very little, and have a near tanning action as is shown by the fact that the treated dry skin is flexible. The last 2 acids (Nos. 15 and 16) repress swelling, raise Ts, and stabilize the skin structure even more. Salts of acids Nos. 4, 6, 7, 10, 11, 12, 13, and 14 show lyotrope swelling. With salts of acids with increasing dipole effect Ts decreases and hide structure is loosened, but with salts of acids with 3 or more rings (No. 15 or 16) there is no effect on swelling or Ts. If the ionic groups of aromatic compounds are not bound by electrostatic attraction to oppositely charged amino groups of the skin, they cause lyotropic swelling and dissolving of hide substance. As the number of points of combination increases, through dipoles and H bonds, lyotrope action reaches a maximum after which there is cross-binding of the fiber structure and tanning. There is technical interest in aromatic sulfonic acids that are so strongly dipolar that they cause no swelling, but also in many cases the tanner wishes to develop a slight swelling for the first tanning stage. A number of acids are available for this purpose (see table). With these acids it is much easier to control swelling than with the usual pickle systems. I.D.C.

Preliminary Report on the Mechanism of Leather Drying. H. Freudenberg. *Das Leder*, **8**, 252–53 (1957).—Leather shrinkage, moisture content, and surface temperature as functions of time were measured during drying of box calf leather at 60°C. and relative humidities (RH) of 60% and 20% respectively. The final moisture content and shrinkage of the leather dried at 60% RH were 15% and 14%, and at 20% RH they were 6.5% and 19% resp. During drying at 20% RH the temperature of the leather surface was constant at 38°C. for a half-hour, then rose rapidly. At 60% RH surface temperature at first rose rather rapidly to 49°, then was constant for about an hour before rising again. Shrinking stopped when the moisture content of the leather was

26–27%. This is near the hygroscopic point, so water at moisture contents below and above this point is held by the fibers in different manners. The amount of shrinkage does not depend on the final moisture content of the leather but mainly on conditions during the early stages of drying. Speed of drying is an important factor in shrinkage. I.D.C.

Uncommon Inorganic Tannages. H. P. Chakravorty and H. E. Nursten. *J. Soc. Leather Trades' Chemists*, **42**, 2–22 (1958).—The action of 31 inorganic compounds on bated calfskin was evaluated on the basis of appearance after drying of the treated hide and on shrinkage temperature (Ts) measurements. Three examples, $CuSO_4$, $AgNO_3$, and $Hg(C_2H_3O_2)_2$, show that for each metal the pH of maximum absorption by the pelt is near that which gives the highest Ts. Almost without exception, the precipitation point of a compound lies near the pH at which its solutions give tanned hide pieces of maximum Ts. For high tanning power this pH does not need to be near the isoelectric point of collagen. The use of complex salts produced results less favorable than those obtained with normal salts. Real tanning power can be associated only with compounds of Hg, Al, Zr, C, Si, P, Cr, Fe, and possibly Va, W, and Co. Arrangement of metallic ions in order according to the maximum Ts which they impart to collagen provides a series similar to that constructed from the stability constants of the metallic complexes of amino acids. J.M.C.

Potentiometric Titration Studies and Tanning Action of Aluminum Complexes. D. Williams-Wynn. *J. Soc. Leather Trades' Chemists*, **42**, 22–31 (1958).—The effect of addition of acetate, formate, lactate, tartrate, and citrate ions to solutions of sodium aluminate was studied by the potentiometric titration technique. Even at ratios as high as 6 equiv/mol Al, little useful masking action was found with formate and acetate. Tartrate, lactate, and citrate stabilize Al solutions against precipitation at ratios of 2 equiv/mol Al. Equal molar combination seems to occur between tartrate and Al in the formation of complexes, while citrate and lactate form complexes in which the ratio of masking agent to Al varies. Tanning with the complexes was evaluated by assessing the leatheriness of the products, by measuring shrinkage temperature (Ts), and by determining resistance of the product to wetting and drying. Most stable leather with highest Ts is obtained with formate, acetate, and lactate masking, while citrate and tartrate complexes give very unstable leather of low Ts. J.M.C.

Role of Proline in Polypeptide Chain Configuration of Proteins. A. G. Szent-Gyorgyi and C. Cohen. *Science*, **126**, 697 (1957).—The generalization is suggested that less than 3% proline distributed statistically in a chain permits more than 50% alpha helix, about 8% proline deforms the backbone into a random coil, and a very high content of proline may favor the poly-l-proline-type helix. R.M.L.

Potentiometric Determination of Sodium Sulfide. J. Gallus. *Chem. Anal. (Poland)*, **2**, 249–57 (1957) (English summary); *Chem. Abstr.*, **52**, 1855*i*.

Tannin-like Substances. II. The Formation of Purpurogallincarboxylic Acid by Enzymic Oxidation of Gallic Acid. K. Herrmann. *Arch. Phar.*, **287**, 497–503 (1954); *Chem. Abstr.*, **52**, 2115*i*.

Differences in the Fine Structure of Collagen and Reticulin as Revealed by the Polarizing Microscope. D. B. Brewer. *J. Pathol. Bacteriol.*, **74**, 371–85 (1957); *Chem. Abstr.*, **52**, 2124*f*.

Structure of Proteins. III. Determination of C-Terminal Amino Acids in Proteins. E. Scoffone, A Turco, and M. Scatena. *Ricerca Sci.*, **27**, 1193–98 (1957); *Chem. Abstr.*, **52**, 2135*a*.

What the Shoe Industry Demands of Leather. W. Hermanns. *Das Leder*, **8**, 161–66 (1957). **Sole Leather (Factory-Type).** H. Bube. *Ibid.*, 166–68. **Sole Leather (Hand Worker–Type).** H. Jüngst. *Ibid.*, 168–70. **Upper Leather.** H. Freudenberg. *Ibid.*, 170–71. **Discussion.** *Ibid.*, 171–72.—Reports of 4 talks at the annual VGCT meeting in Trier. The shoe manufacturer must meet the wishes and demands of those who wear his shoes, and his requirements will vary with the type and price level of his product. Some unsolved problems are discussed. Embossed leather must be hard and firm in order to hold the pattern, but at the same time it should be flexible for good lasting. Embossing covers defects so that sorting is difficult, and the defects may reappear during lasting. Shrunken leather is soft and flexible, but necks should never be used for this type of leather. Box calf leather should be firm, strong, and have a fine grain and brilliant appearance; in fact the shoe manufacturer wishes to have the uniformity of color of a casein pigment finish combined with the beautiful color and grain pattern of an aniline finish. A coating that could be easily removed would be very useful for preventing smudging in the shoe factory; foil is used in some factories but it is quite expensive. Movement of tannin or fillers from sole leather to the shoe uppers by moisture is the cause of stains, spue, and much trouble. Many waterproofing treatments for sole leather have been proposed, but none are satisfactory. They change the color of the leather, interfere with its natural properties such as breathing, and require expensive equipment for application. Custom shoemakers use 30% of the total sole leather production. They use high-quality sole leather because wear and other factors are of more importance than price. Old, long-time tannage, with no loading, produced excellent leather, but the demand is now for more flexible leathers. A leather-testing method is needed that will enable the shoe manufacturer to evaluate leather from the viewpoint of the last user, and one that is simple and intelligible to a nonchemist. Present specifications should be reexamined and revised. I.D.C.

The Development and Operation of a Purification Plant for a Tannery. L. Feikes and E. Röth. *Das Leder*, **8**, 114–17 (1957).—Measurements of quantity of sewage from a tannery and analytical data were accumulated over a period of months. The sewage characteristics were as follows: pH over 10, reducing action in the cold because of the presence of sulfide and

polysulfides, a permanganate requirement 3–5 times that of domestic sewage, high viscosity and specific gravity, gray-brown color, cloudiness, and presence of weighable amounts of sediment even after settling for several hours. The previous method of clarification had consisted of settling for 2 hr., but this did not change pH, color, turbidity, or odor. Hourly variations in volume, pH, solids, and sulfides are shown in curves. It was concluded that chemical treatment would be effective only on the basis of the total daily volume. Three reactions are important: precipitation of sulfides, decrease of alkalinity, and flocculation of colloids. The clarifying agent must have cations that will form difficultly soluble sulfides at pH 7 or above, anions of mineral acids, and ions that will form complexes with proteins and their decomposition products. These requirements are best met by iron salts of mineral acids. Numerous tests were made with $FeSO_4$ and $Fe(HCO_3)_2$; the latter was prepared by treating iron with CO_2 and air in aqueous solution. Dosage of iron salt should be based on the H_2S content of the sewage. Precipitation was rapid and complete only if the sewage was neutral. However, if sufficient iron solution was added to lower the pH value to 7.0, the excess iron in solution slowly oxidized over a period of days and precipitated; this was not economical nor expedient. Much better results were obtained with $Fe(HCO_3)_2$ solutions which contained excess CO_2. If only a slight excess of iron was added, the excess rapidly oxidized to $Fe(OH)_3$ and precipitated within 2 hr. $Fe(OH)_3$ in the nascent state is known to be an excellent flocculating agent for colloidal protein. The excess CO_2 precipitated the lime, and these simultaneous reactions resulted in a heavy residue that settled rapidly. The supernatent liquid was clear, bright, colorless, odorless, neutral, had no reducing action in the cold, and did not further precipitate. The process was independent of sewage concentration to a large degree. The amount of solids in the sewage was reduced by this treatment from, for example, 5500 mg. to 2500; neutral salts could not be removed economically with commercial agents. The clarified sewage had a small permanganate requirement, indicating that the treatment removed only ionic substances or those with polar groups and that the remainder could only be removed biologically. The above results indicated it would be preferable to combine the $FeSO_4$ and $Fe(HCO_3)_2$ methods. The amount of iron and acid to be added in the treating solution should be calculated from data on sulfide content and alkalinity of the sewage. Then the proper proportions of sulfate and bicarbonate must be determined experimentally for the particular sewage to be treated. A description of a plant for treating sewage by this method is given. The sewage is passed through a screen and sand filter to a mixing (equalizing) tank that holds a half-day's production. A constant amount is drawn off continually to a precipitation tank where the neutralizing agent is added, then it moves to a settling tank where it remains for 6 hr. The sludge is pumped from the settling tank to a drainage bed where it is dried. The $Fe(HCO_3)_2$ solution is made by pumping some of the clear effluent into the top of a tower filled with iron chips. Flue gas from the powerhouse is passed up through the tower. Cost of treatment is estimated as 6.46, and development and amortization at 6.81 German cents per cubic meter.

I.D.C.

Hides and Leather in Ancient Rome. G. A. Bravo. *Cuoio, Pelli, Mat. concianti*, **33**, 391–421 (1957).—The first accounts of leather manufacture in ancient Italy are those of the Etruscan and Sardinian peoples about the 7th

century B.C., and early in the 6th century there is some mention of tanners' corporations. In classic Roman times leather manufacture was very widespread, and leather was used not only for shoes (more than 22 types are recorded) but also for clothes, mantles, belts, laces, quivers, scabbards, socks, boxes, saddles, cuirasses, shields, tents, etc. According to Diocletian's Edict (A.D. 301) the price of a pair of shoes was 60–150 *denarii* (the value of the *denarius* corresponded to 0.04 g. gold). Tannage was done with fatty matters, alum, or tannins; the tannins were those of oak, valonia, gall nuts, sumac, and chestnut. Very numerous Roman leather remains have been found at Windisch (Switzerland) where the 11th Roman Legion was stationed for about 100 years in the 1st century A.D. to guard the Rhine River frontier. G.A.B.

Researches on the Fixation of Transformation Products of Oils from Marine Animals on Hide Substance. R. Airoldi. *Chim. e Ind.* (Milan), **39**, 283–87 (1957).—These researches were undertaken to determine if the oil tannage is due only to the action of acrylic aldehyde and if the fatty matters not extractable with solvents are combined with the hide substance or only mechanically adsorbed. Tanning experiments were made with cod oil (I no. 154, acid. no. 1.98), with seal oil (I no. 149, acid. no. 2.71), and with the fatty acids extracted from both. The tanning method was that of Czepelak (*Oesterreich. Leder Ztg.*, **7**, 89 [1952]). By physical and microscopical observations it was shown that at 48°C. the hide becomes very soft, the grain side is deeply corrugated, and the stretch resistance decreases about 40%. The analyses for the "combined fatty matter" (calculated on a defatted basis) gave: tanning with cod oil, 7.52%; with fatty acids of cod oil, 8.86%; with seal oil, 4.27%; and with fatty acids of seal oil, 6.22%. The leathers tanned with the oils had shrinkage temp. 60°C., and those tanned with the corresponding fatty acids, 53°–54°C. The tannages carried out with acrylic aldehyde, either in 0.66*M* solution in 60% alcohol for 24 hr. (following Gustavson) or in H_2O solution (0.1–0.4%), or even with cod oil containing 0.3 to 1% acrylic aldehyde, gave leathers showing a shrinkage temp. of 82° in the first case, and of 61°–72° in the others. Only about 0.5% aldehyde is fixed by the hide. The oil tannage seems to be due not only and not always to the action of acrylic aldehyde but also to the action of other transformation products of the oils. G.A.B.

The Properties of Leather from the Scientific Viewpoint. W. Grassmann. *Cuoio, Pelli, Mat. concianti*, **33**, 532–39 (1957).—Cooperation between representatives of scientific research on leather and publicity personnel is necessary to permit the latter to set forth the properties and advantages of leather on a scientific basis. This cooperation must take into account the technical processes, methods of testing, and evaluation of the properties. The characteristic biological structure of leather is the cause of its porosity, and no other material possesses it. The surface tension, which increases when the diameter of the pores decreases, hinders the transfer of liquids through the pores, but not that of gases; thus a good leather is permeable to gases and vapors and nearly impermeable to water. Many other properties are of course due to the inner reticular structure of leather, i.e., the great internal surface, the flexibility, and the high thermal-insulating power. Plastic deformations undergone by leather are not reversible. Strength and stretch are influenced by the methods

of tanning and finishing. From the chemical standpoint it is to be pointed out that the protein fibers are rich in polar groups (this is a marked difference from many other fibers made with polymerized materials), and this fact permits leather to bind high amounts of water vapor. These properties, and several others which may be required for leather employed for special purposes, may be imparted by suitable methods of tanning. Testing and controlling the manufacture of leather and leather wares are the main problems that leather researchers must solve to improve quality and help the work of the publicity people. G.A.B.

Chemical Washing of Boilers. M. Zanazzi *Ind. saccarif. ital.*, **50**, 74–83 (1957).—Chemical washing is noticeably less expensive than mechanical cleaning. It is necessary to give first an alkaline wash (with $NaOH+Na_2CO_3$) to dissolve salts which are insoluble in acids, then a chemical wash with inhibited acid. A steel tube in contact with 5% HCl without inhibitor undergoes a loss of 1.5 g. per sq. m. per hour, while in the presence of 0.5% formaldehyde it shows a loss of 0.34, and with 0.5% molasses a loss of 0.30 g. per sq. m. per hour. However it is sufficient to use only 2% acid, increasing the concentration of the inhibitor even to 5%, and following with a washing with diluted solution of Na_2CO_3. G.A.B.

Methods of Determination of Water Solubles in Leather. E. Simoncini. *Cuoio, Pelli, Mat. concianti*, **33**, 460–68 (1957).—The various methods of determination of H_2O-soluble matter in vegetable-tanned leather do not always give results agreeing with one another. Comparative analyses made with the International Shake Method, Swiss Method, Koch Extraction Method, and ALCA Method show that the last gives the highest results. A complete extraction is never obtained even with a very long treatment of the leather sample, but with 7 successive extractions not more than 1% of soluble matters may remain in the leather. The specifications of the above-mentioned methods allow the extraction of 51.16% to 76.62% of the presumable total soluble matter contained in the leather. Some experiments were made to examine the influence of temperature, duration of digestion, and shaking, and it was observed that by slight variations of the original methods it is possible to obtain results which are rather concordant. These variations are: International Method: extraction at 50°C.; Swiss Method: ashing the residues without addition of H_2SO_4; ALCA Method: extraction of 10g. leather at 20°C. with previous digestion of the sample in water for 17 hr.; Koch Method: no change. G.A.B.

Limits of Reliability of the Physical Tests of a Rapidly Tanned Sole Leather. E. Simoncini. *Cuoio, Pelli, Mat. concianti*, **33**, 358–89 (1957).—Leather is not homogeneous, and generally some changes in physical properties are found according to the place of sampling; the causes of the poor uniformity are mainly the differences in thickness and specific weight of the fibers and the different orientation of the fiber bundles. Sometimes it may happen that the physical properties determined on few samples appear to be abnormal, while the chemical analysis, on the contrary, may show perfectly regular results —the abnormality originating from the excessively low number of samples in

the physical determinations. To know the real range of changes that properties of a leather may undergo over its whole surface, some physical determinations were made on a single sole leather, with the following max. and min. results: thickness, 3.78–5.55 mm.; same after wetting, 4.11–6.06 mm.; sp. gravity, 1.09–1.24; same after wetting, 0.91–1.7; strength, 1.5–3.3 kg. per sq. mm.; stretch, 14–26%; hardness (% penetration of a steel cylinder of 4-mm. diameter, under a pressure of 70 kg.), 80.0–89.4; same after wetting, 46.9–65.5; same after reconditioning, 62.7–77.5; rigidity (Olsen apparatus), 1.56–3.02; wear index (% of decrease in thickness after 100 revolutions of the wheel, British apparatus), 0.66–2.27; water permeability coeff., 0.39–1.01; water absorption after 1 hr., 14–25%; same after 2 hr., 19–32%; same after 24 hr., 31–47%; air permeability coeff., 0.016–0.046. Notwithstanding the differences between max. and min. values found, the data obtained, when examined with the aid of chemical analysis, may give sufficient information for evaluation of a leather. G.A.B.

Pigskin and Pig Leather. F. Stather. *Cuoio, Pelli, Mat. concianti,* **33,** 435–42 (1957).—Pigskin was once used only in small amounts for manufacture of case leather and other less common goods, but after World War II it assumed a greater importance, particularly after the researches carried out by the Freiberg Leather Institute. Contrary to common opinion, pigskin is not very rich in fatty matters; grease exists only in the subcutaneous layer in funnel-shaped hollows and around the roots of the bristles. The main difference between pigskin and the skin of other mammalia is in the connective elastic tissue which is particularly developed in the upper layer immediately below the grain and around the holes of the bristles. The triple disposition of the follicles is characteristic: in a unique grease papilla there are three bulbs of bristles growing together. The scalding of pigskins with water over 55°C. to remove the bristles causes damage to the fibers and particularly to the grain, together with a decrease in strength and enzyme resistance. The better skins are those of males, weighing about 3.2 kg. It is recommended that pigskins be soaked for 1 day at 18°–20°C., that excess grease be removed mechanically, that the skin then be unhaired with a lime bath containing 4% sulfide in a drum for 8 hr. at 28°–30°, followed by a pure lime bath for 4 days. Bating is done with 1% of a good commercial bate for 1 hr. at 36°C. maximum. Special defatting is not necessary; about 50% of the grease is removed during liming and about 10–20% during deliming and bating. Chrome tanning is done as usual, but it is suggested that 0.25% of a dye be added to the chrome bath in order to follow the penetration easily. Retannage with vegetable or synthetic tannins is necessary to avoid an eventual excess of elasticity and to give a better feel. Fatliquoring is done with little grease (with a low emulsifiability index) emulsified with water; fluffing is done on the grain side when the leather is still wet. The bottom season is preferably prepared with a soft synthetic wax, and the following 3–4 seasonings are made with albumin and polymerized resins in such a way that the softness of the various layers decreases upwards. The glaze after dyeing, the marking of a finer grain after spray-dyeing, and the eventual new seasoning prevent the skin pores from protruding. For the manufacture of suede use of a weak pickle and basic chrome liquor is suggested; masking salts are inadvisable. Fatliquoring must be done twice separately, 2/3 before and 1/3 after splitting. G.A.B.

Recent South African Developments in Rapid Sole Leather Tannage. S. G. Shuttleworth. *Cuoio, Pelli, Mat. concianti*, **33**, 442–59 (1957).—Two rapid-tannage processes using only wattle extract are described. *Osmotan process:* the unhaired hides are pickled in H_2SO_4 (about 1.5% on limed-stock weight) + 10% salt for 1 day, then placed in tan liquor at pH 3.0 and 25° Bark. where only the surface is tanned. Deliming is not complete after 1 day in pickle but becomes complete during the surface tannage. Four tan pits follow, at 60°, 67°, 74°, and 80° Bark. resp., while pH values rise from 3.1 to 3.3 (a constant value is obtained by adding small amounts of H_2SO_4, but a strength control is always necessary); the hides remain 1 day in each pit and the countercurrent system is used. Bellies and shoulders may be tanned by the same method, but the density of the liquors is only 20° Bark. in the grain tannage pit and increases to 52° Bark. in the 4th pit. *Liritan process:* after liming 48 hr. and 1 day in the unhairing drum with straight lime liquor, the hides are pickled with 1.25% H_2SO_4, 5% salt, and 2.5% phosphate. The surface is tanned in a pit at 20° Bark. and pH 3.0. The following pits contain wattle extract at 67°, 74°, 81°, 88°, 94°, and 100° Bark., with pH values from 3.0 to 3.3 (pH is adjusted by modifying acid added to pickle), and the hides remain 1 day in every pit. Bellies and shoulders are tanned in 6 pits at concentrations increasing from 15° to 60° Bark. The hides are then transferred to a drum with powdered wattle extract and fillers, and drummed until the material is absorbed. The advantages of the method are mainly: deliming and pH corrections are made with low priced mineral acid; maximum value is obtained from small amounts of phosphates; complete utilization of the tan liquor is achieved; at pH 3.0 the oxidation of tannin is minimized. G.A.B.

Determination of the Active Power of Chemical Disinfectants by Measuring Electrical Conductivity. A. Simoncini. *Cuoio, Pelli, Mat. concianti*, **33**, 67–87, 181–202, 265–80 (1957).—The use of compounds having bactericidal or bacteriostatic activity is widespread in the tanning industry because hides and skins are easily putrefiable. In order to know the optimum conditions of their activity (concentration, temperature, time, etc.) it is necessary to determine the efficiency of every disinfectant, and several methods have been previously proposed. The determination of the Rideal-Walker coefficient, or phenol coefficient, measures the activity of a given compound in comparison with pure phenol against a suspension of microorganisms, but this method is of little importance when many microorganisms are acting together; however much improvement is obtained with the modification recently proposed by Hausam (*Leder*, **4**, 25 [1953]). Several other methods deal with the quantitative determination of the degradation products of the proteins or with bacterial count, and they are evidently very long and tiresome and give hardly reproducible results. Other methods by which external properties are examined (odor, plumpness) may give only some orientative but not exact results; and in general all the methods suitable to give sure values, even those recently proposed (e.g., paper bacteriography), need the help of a bacteriologist to measure the increase of the number of microorganisms in the material to be examined during the aging of the cultures prepared following some specified prescriptions. A new method is now proposed, based on conductivity measurements. A nonsterile solution of gelatin or of another N-containing macromolecular organic compound shows an increase of its electrical conductivity even a few hours

after the preparation, due to the growth of microorganisms which decompose the protein giving products with less complex molecules. If a disinfectant is present, this increase of conductivity is stopped or retarded. In these experiments a 0.7% solution of gelatin was prepared and tested with some commercial products together with several strong disinfectants (pure chemicals: phenol, *p*-nitrophenol, Na pentachlorophenate, $HgCl_2$). The curves "electrical conductivity vs. time" drawn for every product at increasing concentrations show the limit concentration corresponding, in the condition of the experiments, to the maximum of activity. The curve in these conditions is nearly parallel to the axis of the abscissae. The limit concentrations observed are: 0.175% for phenol, 0.015% for Na pentachlorophenate, 0.005% for $HgCl_2$, 0.012% for *p*-nitrophenol, and 0.01 to 0.04% for the examined commercial products. Several tests were made with this new method in comparison with the determination of phenol coefficient and with the bacterial count. These latter methods do not indicate the actual capacity of the disinfectants to hinder or to stop the degradation of the protein, and therefore it is suggested that in ordinary laboratory researches the bacteriological method be supplemented or even replaced with the conductivity method. This procedure may even be applied in other industries, operating on different substrates and in the presence of qualitatively and quantitatively known microorganisms. G.A.B.

PATENTS

Tanning Compositions. Brit. Pat. 785,104. Farben-Fabriken Bayer Akt.-Ges. Appl. Jan. 19, 1956.—A mixture of organic acids comprising at least 50 per cent of glutaric acid is incorporated in a powdered synthetic tanning agent to adjust the acidity thereof. A suitable mixture of acids is that obtained as the by-product of the oxidation to adipic acid of cyclohexane, cyclohexanol, cyclohexanone or a mixture thereof, and may comprise glutaric, succinic, adipic and oxalic acids. The mixture of acids which is preferably present in an amount of 5—20 percent by weight of the tanning agent, may be incorporated after the latter has been dried to a powder, or before; in the latter case the mixture may be spray-dried. Examples are given in which the tanning agent is prepared by (1) reacting an *o*-dihydroxy benzene derivative with an aromatic compound containing sulphonic acid groups and an aldehyde and condensing with formaldehyde (German Specification 870,268); or (2) condensing a dihydroxy-diphenyl sulphone not having sulphonic acid groups with formaldehyde in the presence of an aromatic sulphonic acid or formaldehyde or sulphuryl chloride condensation products thereof (German Specification 611,671). The mixtures have a *p*H of about 3.3.

Treatment of Hides. Brit. Pat. 785,112. Burton, D., Reed, R., and Flint, F. O. Appl. Feb. 18, 1954.—A composition capable of modifying the mucoid material in skins and hides comprises 0.025—50 per cent of a commercial preparation of a carbohydrase or mixture of carbohydrases and a buffer solu-

tion in the *p*H range 3 to 12.5. In the Provisional Specification the enzymes are referred to as mucolytic enzymes. Enzymes specifically referred to are polygalacturonidase, elastase (preferred *p*H 8.9), pectinase (preferred *p*H 4.0), hyaluronidase, chondroitinase and diastase or amylase (preferred *p*H 6.5). 0.025—5 per cent of a commercial preparation of one or more proteolytic enzymes, e.g. trypsin and papain, may be present in the composition initially or may be added during the treatment. The compositions may be used at any stage in the treatment of hides or skins, e.g. as follows: (1) *for soaking back* fresh, dried, salted, limed or otherwise preserved hides or skins, if desired after a conventional soaking-back operation with or without alkalies, wetting agents or bactericidal agents; for this purpose the buffer solution preferably has a concentration of 0.25 to 10 per cent by weight and a wetting agent may be added: conditions are generally similar to those described in (2) below. The hides may if desired be left in this bath to effect depilation and/or loosening and/or bating; (2) *for removing keratinous covering structures* such as hair at lower *p*H's than are usual, preferably at a temperature of 15° to 35° C.: aeration may be employed and the treatment may be from 2 hours to 7 days or more in a drum, paddle, pit or vat: the hair &c. may be detached or may have to be pulled. Further enzymes may be added to the solution during the treatment and activators such as sodium chloride, sodium sulphate, potassium chloride and calcium chloride may be employed, e.g. in concentration up to 10 per cent by weight. Suitable procedures are to soak the hides in the solution and allow either to stand in piles at 15—35° C. to hang them individually, or to "paint" a composition thickened e.g. with wheat or other flour, starch, bran or gelatin in a concentration of up to 90 per cent by weight and containing a buffer solution of concentration within the range 0.25 to 10 per cent by weight of the composition on the flesh side of the skins, pile or hang, and pull the hair &c. (3) *For loosening* the fibrous structures of the corium in like manner, in place of liming or bating or even after conventional tanning or partial tanning. When treating tanned stock the *p*H is preferably between 3 and 8 to avoid stripping or overneutralizing a vegetable or chrome tan. This assists in the subsequent even absorption of dye or fat liquor. (4) *For pre-treatment of skins and hides to be used in the production of parchment and rawhide* products in which case the usual liming treatment is unnecessary. (5) *For tanning* in the presence of vegetable tanning agents such as myrobalans, preferably at *p*H of 3 to 10; naturally buffered pectinase/myrobalans and pectinase/quebracho compositions are described in Examples (19 and 20). (6) The process may be applied *after tanning* to material which has been dyed and/or fat-liquored and/or finished. Examples are given, which in addition to the abovementioned agents and activators listed below refer to: alkylphenylpolyglycolether wetting agents in soaking and depilating baths (cetyl pyridinium bromide and sodium salt of dodecyl benzyl sulphite are also mentioned as wetting agents of cationic and anionic types); loosening of chromed and vegetable retanned leather; as buffer solutions, acetic acid/sodium hydroxide, sodium acetate/acetic acid, glycine/sodium hydroxide, borax/boric acid, potassium dihydrogen phosphate/sodium hydroxide, boric acid/sodium hydroxide and sodium formate/formic acid are referred to. Suitable activators for the enzymes are phosphate, citrate, formate, borate, iodide and hydrosulphite anions, calcium, copper, zinc and chromium cations, hydrogen peroxide, simple sugars such as galactose, dextrose and maltose, disaccharides such as sucrose; starch, yeast, bran and flours. Calcium pyrophosphate and potassium iodide are specified in examples.

Leather Diaphragms. Brit. Pat. 785,113. United Gas Industries, Ltd. Appl. Dec. 20, 1954.—Leather intended for diaphragms is improved with respect to flexibility and length of life by treatment by impregnation with an emulsion or dispersion of at least one monomeric ethylenically unsaturated compound, e.g. acrylonitrile, ethyl methacrylate and unsaturated polyesters, which polymerizes upon the fibres and fibrils of the structure of the leather. The treatment may take place after the fatliquoring of the leather.

Process for the Production of Artificial Leather Having Suede-like Character. U. S. Pat. 2,811,464. Karl Stiehl and Karl Reus, Hanau (Main), Germany, assignors to Deutsche Gold- und Silber-Scheideanstalt vormals Roessler, Frankfurt am Main, Germany. Appl. Dec. 12, 1955.—1. A process of producing suede-like artificial leather which comprises applying a thin coating of a pigmented plasticizer containing polyvinyl chloride paste over a pliable gelled polyvinyl chloride coating on a base, dusting a thin coating of a powdered blowing agent which gasifies at a temperature between 100 and 200° C. on the polyvinyl chloride paste coating, and heating the polyvinyl chloride paste coating carrying the dusted on blowing agent to a temperature at which the polyvinyl chloride paste gels and the blowing agent gasifies.

Shrinkproofing of Wool with Epoxy Resins and Polyalkyleneimines. U. S. Pat. 2,817,602. Clay E. Pardo, Jr., Albany, Calif., assignor to the United States of America as represented by the Secretary of Agriculture. Appl. Sept. 18, 1956.—1. A process for shrinkproofing protein fibers which comprises impregnating the fibers with an emulsion containing a polyalkyleneimine and an epoxy resin and thereafter curing the impregnated fiber at a temperature from about 50 to about 100° C.

Protective Covering of Leather. U. S. Pat. 2,823,157. Raymond P. Hofferbert, Beverly, John P. Szumski, Salem, and William L. D. McKay, Marblehead, Mass., assignors to United Shoe Machinery Corporation, Flemington, N. J., a corporation of New Jersey. Appl. Jan. 21, 1953.—1. The method of protecting leather which comprises the three step process of first disposing a thin sheet of high molecular weight polyethylene on a surface of a leather sheet and heating the polyethylene sheet and substantially said surface only of the leather sheet to a temperature near but below the melting point of the polyethylene, secondly pressing a heated surface simultaneously against all areas of the sheet of polyethylene to be joined to said leather surface to melt the polyethylene to fluid condition and to force the hot fluid polyethylene into the surface of the leather, said heated surface having a temperature substantially above the melting point of the polyethylene, and thirdly cooling the heated surface promptly after establishing pressure to reduce the temperature of the surface to a point well below the melting temperature of the polyethylene and thereafter releasing said pressure.

Lost Circulation. U. S. Pat. 2,811,488. Alfred C. Nestle, Houston, Tex., and Victor J. Tronolone, Downey, Calif., assignors to The Texas Company, New

York, N. Y., a corporation of Delaware. Appl. June 28, 1954.—1. A composition of matter suitable for use in a drilling fluid for correcting lost circulation comprising an admixture of fibrous, ground leather and Ottawa sand having a size range in the range 20–30 mesh, said fibrous leather and said sand being present in the admixture in the weight ratio range 1:7–9, respectively.

Method of Leather-Fatting. U. S. Pat. 2,822,235. Rudi Heyden, Dusseldorf, Gerhard Dieckelmann, Dusseldorf-Himmelgeist, and Jürgen Plapper, Dusseldorf-Holthausen, Germany, assignors to Böhme Fettchemie G. m. b. H., Dusseldorf, Germany, a corporation of Germany. Appl. Nov. 9, 1954.—1. The method of retarding discoloration of leather on exposure to light which comprises fatting said leather by applying to said leather an aqueous emulsion of an emulsifier and a compound selected from the group consisting of epoxidized derivatives of high-molecular unsaturated fatty acids and high-molecular unsaturated fatty alcohol esters of monobasic and polybasic acids.

Artificial Leather. Brit. Pat. 784,190. Deutsche Gold- und Silber-Scheideanstalt vorm. Roessler. Appl. Dec. 2, 1955.—Artificial leather having a fine pore surface is produced by coating a substrate, e.g. woven or knitted fabric, paper or fibre, with a thin layer of colored polyvinyl chloride paste containing a plasticiser, e.g. dioctyl or dibutyl phthalate, spraying on to said layer a powder which gasefies at a temperature between 100° and 200° C., e.g. ammonium carbonate or bicarbonate, and heating the coated material to completely gel the polyvinyl chloride paste and gasefy the powder. The artificial leather may be lightly buffed.

Impregnated Fabrics for Shoe &c. Stiffeners. Brit. Pat. 783,776. Rhenoflex Ges. Appl. Jan. 15, 1954.—Stiffeners, e.g. for leather toe-caps, consist of a fabric, felt, or fibre-fleece impregnated with a water-soluble, hardenable, precondensate of a synthetic resin, and also with an aqueous dispersion of a polymer of vinyl chloride or acetate or of an acrylic or methacrylic ester, so as to incorporate 60–95 per cent of the pre-condensate and 5–40 per cent of the polymer. Urea- or malamine-formaldehyde pre-condensates, or a phenol-aldehyde resol, may be used. Fillers such as wood meal, cotton flock or linters, silica or alumina gel, or kieselguhr may be added. A coarse mesh fabric may be incorporated into the carrier or base. To prevent sticking together in storage, the impregnated material may be coated with methyl cellulose. In an example a mixture of an aqueous solution of a urea-formaldehyde condensation product, an oleo-resin, and kaolin is rubbed through a funnel mill and mixed with an aqueous dispersion of polymethyl acrylate containing ammonia. A coarse mesh fabric is impregnated with the mixed dispersion and dried. Stiffeners are stamped out from the product, and, before use, are immersed in a solution of ammonium chloride which acts as a hardener for the urea-formaldehyde condensate.

Agglutinated Fibrous Sheet Materials. Brit. Pat. 782,952. Du Pont de Nemours & Co., E. I. Appl. Aug. 12, 1955.—A leather-like product comprises, by weight, 40 to 85 per cent of matted oriented fibres of a synthetic linear condensation polymer and, distributed therethrough, 60 to 15 per cent of a polymeric

binder, chemically dissimilar to the said linear condensation, polymer, incompatible therewith and melting below the softening temperature thereof, the said product containing a volume per cent of interconnecting void space distributed therethrough at least equal to 3000 (weight per cent fibre)$^{-1}$—30 and not greater than 70 per cent. The product may be prepared by mixing the fibres with the binder and then partially consolidating the mixture under controlled conditions of temperature and pressure. Mixing may be carried out by impregnating a porous non-woven mat, web, or felt of the fibres with a solution or dispersion of the binder or, where fibrous or particulate forms of the binder are used, by mixing such as is achieved in conventional carding procedures. The mat, prior to impregnation with the binder should have a bulk density of less than 0.5 g/cc. Mats may be formed by paper-making techniques, carding or deposition from an air stream. Specified polymers for use as the fibre component are dibasic acid/diamine or amino acid polyamides, the dibasic acid/diol or hydroxy acid polyesters or the intermixed polyester/polyamide products or mixtures thereof; such as polytetramethylenesebacamide, polyethyleneterephthalamide, poly-D-, or L-, or DL-alanine, poly-D, or L-, or DL-leucine, and polyepsilon-amino-caproic acid, polydodecamethylene adipate, polyethylene sebacate, polyhexamethylene oxalate, poly-D-, or L-, or DL-lactic acid, poly-D- or L-, or DL-mandelic acid and polyepsilon-hydroxy-caproic acid. Structural fibres ranging in length from 0.01 inch to 8 inches may be used. Specified polymers for use as the binder are cellulose acetate, cyanoethyl cellulose, polyethylene, polypropylene, polyisobutylene, polybutene-2, polyisoprene, vinyl chloride, 2-3-dichlorobutadiene, vinyl acetate, butadiene, styrene, tetra-fluoroethylene/ethylene, vinyl chloride/vinyl acetate and vinylidene polymers and copolymers including both the monoene and diene types. The fibre-binder ratio may vary, on a weight basis, from 40/60 to 85/15. In an example 2 mats of polyhexamethyleneadipamide fibres are impregnated with a dispersion of polyvinyl chloride/di-2-ethylhexyl phthalate in a tetrahydrofuran/dimethyl formamide solvent mixture, dried, plied together and then pressed for 10 minutes at 160° C.

Tanning Hides. Brit. Pat. 778, 153. American Zinc, Lead & Smelting Co. Appl. Feb. 18, 1955.—Hides are tanned by impregnating them with a soluble sulfide or hydrosulphide capable of producing SH ions such as K_2S, NaSH, $Ba(SH)_2$, $Ca(SH)_2$, Na_2S or $(NH_4)_2S$, and then impregnating the hides with an aqueous solution of a zinc salt which reacts with a sulfide or hydrosulphide to fix a zinc compound in the hides. The zinc salts employed are preferably the chloride or sulfate or an ammoniacal solution thereof or a basic salt of the chloride and sulfate. The hides may subsequently be further tanned, for example, with formaldehyde, syntan or chromium salts. According to examples: (I) bated hide was treated in a ball mill with a solution of sodium hydrosulphide and common salt, then removed and ball-milled with a solution containing zinc sulfate and common salt. The hide was finally washed and milled with a solution containing an anionic sulfonated oil and common salt. (III) an aqueous solution of sodium hydrosulphide and calcium chloride was employed to treat calf skin which, after impregnation and draining, was treated with a zinc sulphate solution containing common salt and finally with an aqueous emulsion of an anionic sulfonated oil. A white leather containing 6.4 per cent of zinc was obtained. (V) a hide was treated with sodium hydrosulphide and salt as before and after washing was milled with a zinc sulphate solution containing common

salt. After soaking overnight, a solution of chromium sulphate in water was added and the soaking continued. The hide was finally milled with an aqueous solution of an anionic sulfonated oil. (VI) a hide was treated with sodium hydrosulphide and zinc sulphate as before and a 40 per cent solution of formaldehyde was added to the zinc sulphate solution towards the end of the process. The hide was then washed and treated with an anionic sulfonated oil as before. If additional tanning is required it can be effected at the end of the tanning during the oiling step.

Tanning Leather. Brit. Pat. 779,244. Boehme Fettchemie Ges. Appl. Aug. 10, 1953.—Hides and skins are tanned by applying thereto a water-soluble resinous condensation product obtained by condensing an organic sulphonamide with an oxo-compound or a substance giving off an oxo-compound, and urea, thiourea, guanidine or a derivative of one of these which contains at least one replaceable hydrogen atom, the molar ratio of the oxo-compound to the sum of the other compounds being at least 4:1 (*see* Group IV (a)), and during the application or subsequent thereto insolubilizing the condensation product, e.g. by means of electrolytes, such as formic acid, first runnings fatty acid mixtures or ammonium chloride, or by heating. The tanning may be combined with a fatty tanning, i.e. a tanning with compounds containing a fatty acid radical, or tanning with formaldehyde, with chromium or aluminum salts or with a vegetable tanning. Many fatty tanning compounds are exemplified. Generally the condensates are used in amounts of 2 to 10 per cent by weight of the pelts to be tanned. In examples: (3) bated calf hides are tanned with the condensate of *o*- and *p*-toluene sulphonamides, dicyandiamide and formalin; the hides treated with the condensate in the previous example are further treated with (4) an oleyl alcohol-sodium sulphate paste and finally potassium alum; and (5) a basic chromium tanning agent; (b) delimed bated pelts are pre-tanned with the condensate of Example (3) and a liquor oil emulsified with a cation soap and finally tanned with a vegetable tannin; (7) Vachette leathers are prepared by pre-tanning with the condensate of *p*-toluene sulphonamide, urea and formalin and finally tanned with a vegetable tannin; (8) delimed pelts are pre-tanned with the condensate of *p*-toluene sulphonamide, guanidine and acetone and finally tanned with a vegetable tannin. Specification 748,995 is referred to.

Waterproofing Textiles, Paper, and Leather. Brit. Pat. 778,204. Ciba Ltd. Appl. Dec. 23, 1953.—Hardenable ternary condensates (*see* Group IV (a)) containing COOH or SO_3H group and prepared from (a) an aminotriazine-formaldehyde condensate or ether thereof, (b) an aliphatic acid, alcohol, mercaptan, hydroxy ester, hydroxy ether, amide, urethane, N-methylolamide, or N-methylol urethane, said compound containing a carbon chain of at least 7 carbon atoms and (c) an aliphatic monohydroxy- or monomercapto - monocarboxylic or monosulphonic acid may be used for producing water-repellent effects on textiles, paper or leather. The products are preferably applied in aqueous solution if desired together with aluminium triformate, a zirconium salt, a brine of hydrated zirconium hydroxide, or dispersions of paraffin wax.

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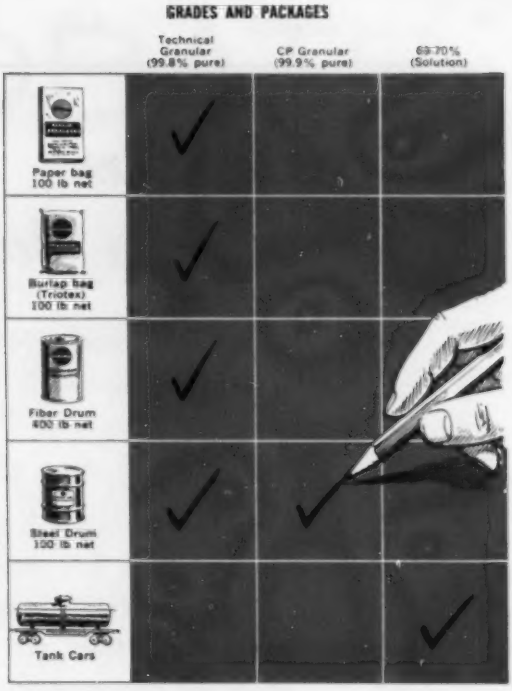
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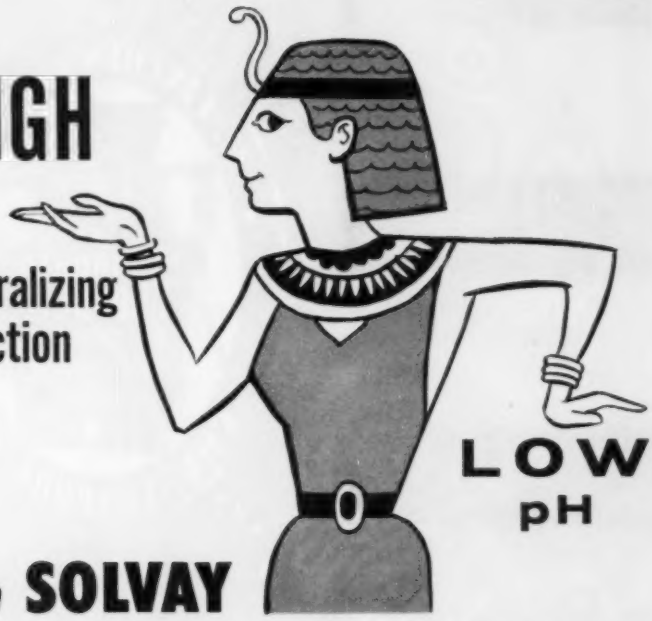

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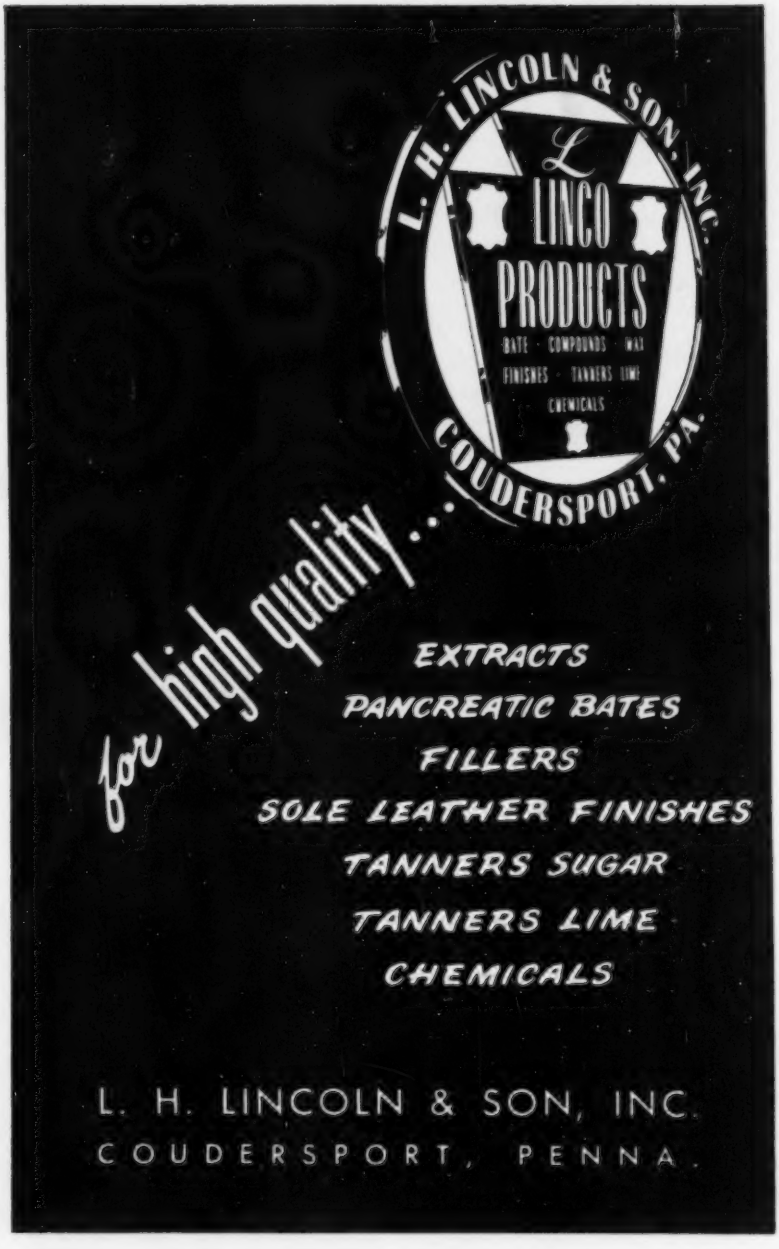
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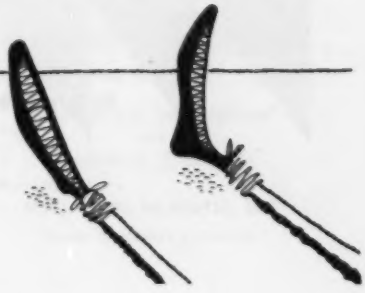

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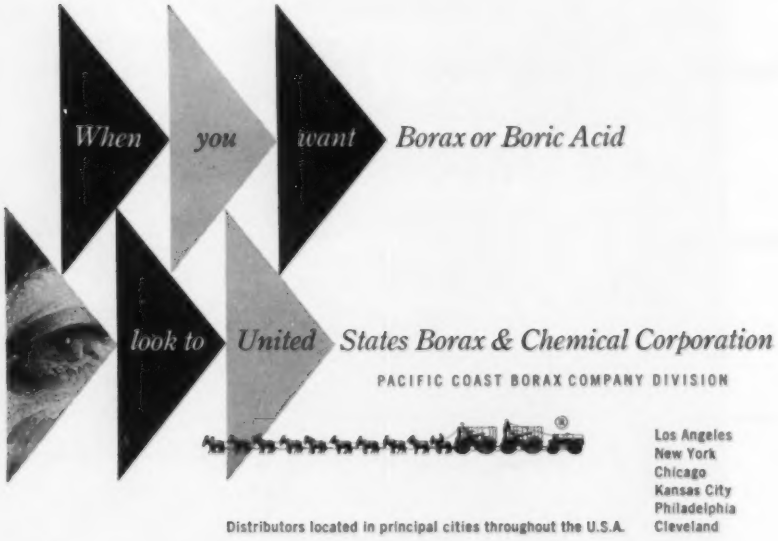

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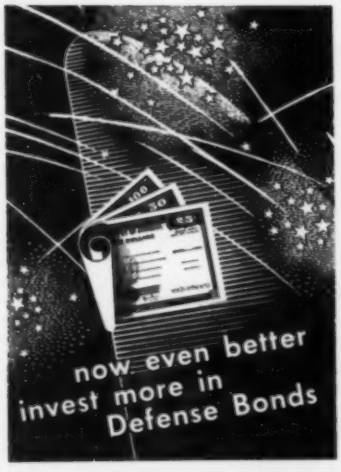

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Split Suede Tanner Colors Directly From The Blue With Synova, Gets Short Silken Nap

Some months ago a progressive eastern split suede tanner wanted to color directly from the blue. But conventional sulfated oils gave him a sheeny buffing job and poor ur.level coloring.

Hearing about Synova and its success on suedes, he made a trial run —following suggestions offered by a Seaboard representative. The results were surprisingly good. So he followed with a full pack—and is now fatliquoring his entire production with Synova.

Stock Upgraded on Table

Coloring directly from the blue, he now produces a low-cost sueded split. And even though his suedes are pasted under fast drying conditions, they are properly lubricated and conditioned—and buff beautifully to a short resilient silken nap. Coloring is improved and much leveler.

By using this fast inexpensive process, he produces a better suede at lower cost. Takes a higher profit margin too. By eliminating pearling, he increases production, using the same equipment.

Also he reports a number of other benefits from using Synova. It is absolutely uniform, has long storage stability, and its odor is clean and pleasant.

Try a Sample Yourself

What about you? Whether you're tanning calf or kid suede, sueded reversible sides or splits, wouldn't it be a good idea to see for yourself what Synova can do for your suedes? Prove what other salesminded tanners are saying — that for pearling, coloring directly from the blue or grain clearing, modern Synova is the finest discovery they ever made. We'd like to send you a trial sample. Write or phone today.

It's time you switched to Synova!

SEABOARD CHEMICALS, INC.

Dept. 5A, 30 Foster St.
Salem, Massachusetts

In Canada: Tennant Union, Ltd., Toronto
Overseas Export: Wolff International, Inc., Milwaukee, Wisconsin, U. S. A.

Mulling over an idea...

Exchanging ideas and considering both the practical and chemical aspects is the first step in planning new products — thoughts preceding the deed. Once we have decided on a plan of action our research department takes over to bring this idea to life.

CHEMTAN COMPANY

EXETER, NEW HAMPSHIRE

All our facilities are devoted exclusively to the tanning industry

CHEMTAN COMPANY EXETER, NEW HAMPSHIRE